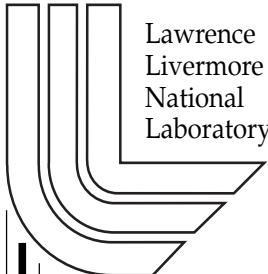


# **Interim Report on Task 1.3: Equilibrium Phase Diagram to Lawrence Livermore National Laboratory for Contract B345772 (11/26/99)**

*D. S. Perera, M. W. A. Stewart, R. A. Day and E. R. Vance*

*U.S. Department of Energy*



**November 26, 1999**

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## **Interim Report on Task 1.3: Equilibrium Phase Diagram**

**To Lawrence Livermore National Laboratory for Contract  
B345772**

D S Perera, R A Day, M W A Stewart and E R Vance

26 November 1999

R99065m

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## 1 SUMMARY

To determine the phase equilibria in the pseudo-ternary systems  $\text{CaHfTi}_2\text{O}_7$ - $\text{Gd}_2\text{Ti}_2\text{O}_7$ - $\text{Al}_2\text{TiO}_5$  and  $\text{CaHfTi}_2\text{O}_7$ - $\text{Gd}_2\text{Ti}_2\text{O}_7$ - $\text{MgTi}_2\text{O}_5$  five compositions containing 10 mol % additions of  $\text{Al}_2\text{TiO}_5$  and  $\text{MgTi}_2\text{O}_5$  respectively, were initially prepared using an alkoxide-nitrate route. They were sintered at  $1300^0$ - $1400^0\text{C}$  in air. The sintered samples were analysed by X-ray diffraction, scanning electron microscopy and energy dispersive X-ray spectrometry. Al and Mg systems have approximately same effects as the binary pyrochlore-zirconolite system. Perovskite was observed in the Al-containing system but was not observed in the Mg-containing system. In the Mg-containing system the phase region of pyrochlore and zirconolite is much smaller than in the Al-containing system. However, only 2M zirconolite (except for a trace of 4M zirconolite in two compositions) was observed in contrast to the previous work on the pseudo-binary system  $\text{CaHfTi}_2\text{O}_7$ - $\text{Gd}_2\text{Ti}_2\text{O}_7$  where 4M zirconolite was also present. The additions of Al and Mg evidently suppressed the 4M zirconolite.. Tentative phase boundaries have been marked for both systems. Further compositions away from the pseudo-binary system  $\text{CaHfTi}_2\text{O}_7$ - $\text{Gd}_2\text{Ti}_2\text{O}_7$  have been prepared to fine-tune the phase boundaries.

## 2 INTRODUCTION

One of the aims of Phase 3 of Task 1.3 is to determine the phase equilibria in the pseudo-ternary systems  $\text{CaHfTi}_2\text{O}_7\text{-Gd}_2\text{Ti}_2\text{O}_7\text{-Al}_2\text{TiO}_5$  and  $\text{CaHfTi}_2\text{O}_7\text{-Gd}_2\text{Ti}_2\text{O}_7\text{-MgTi}_2\text{O}_5$  at  $1300^{\circ}\text{C}$ - $1400^{\circ}\text{C}$ . The phase equilibria for the pseudo binary system  $\text{CaHfTi}_2\text{O}_7\text{-Gd}_2\text{T}_2\text{O}_7$  have been determined previously [1]. Ten mol % additions of  $\text{Al}_2\text{TiO}_5$  and  $\text{MgTi}_2\text{O}_5$  were added to the compositions along the  $\text{CaHfTi}_2\text{O}_7\text{-Gd}_2\text{T}_2\text{O}_7$  join. In this interim report five compositions each for the Al and Mg systems are reported.

## 3 EXPERIMENTAL

### 3.1 Preparation

From the raw materials listed in Table 1, 20 g batches of the compositions listed in Table 2 were prepared by the standard alkoxide-nitrate route [2].

Pellets of calcined precursors ~ 0.5 g and 20 mm diameter were cold pressed at ~ 90 MPa and then sintered on a Pt foil in an  $\text{Al}_2\text{O}_3$  boat in air. The sintering conditions are listed in Tables 3. The heating and cooling rates were  $5^{\circ}\text{C}/\text{min}$ .

### 3.2 Analysis

After sintering, the samples were broken in half; one part was mounted in resin and polished to a 0.25  $\mu\text{m}$  finish and the other part was crushed and examined by X-ray diffraction (XRD) using a Siemens D500 diffractometer with  $\text{Co K}\alpha$  radiation. The polished samples were examined by scanning electron microscopy (SEM) using a JEOL JSM 6300 run at 15 keV and fitted with a Norian Voyager 4 energy dispersive X-ray spectrometry (EDS) for quantitative analysis.

**Table 1: Raw materials used for the precursor batches.**

Element	Raw Materials for Alkoxide-route Batches (raw material, source, catalogue number)
Ca	> 99 % $\text{Ca}(\text{NO}_3)_2\cdot 4\text{H}_2\text{O}$ , Aldrich Chem. Co.; 23712-4
Ti	Titanium isopropoxide, HÜLS Troisdorf Gmb.; 405514
Hf	99.99 % Hafnium n-butoxide, Gelest Inc.
Gd	99.9 % $\text{Gd}(\text{NO}_3)_3\cdot 6\text{H}_2\text{O}$ , Aldrich Chem. Co.; 21159-1
Al	Aluminium sec-butoxide, Merck-Schuchardt; 820054
Mg	>99% $\text{Mg}(\text{NO}_3)_2\cdot 6\text{H}_2\text{O}$ ; Aldrich Chem. Co.; 23717-5

**Table 2: Compositions investigated.**

<b>Composn. Ident. No.</b>	<b>Composition</b>	<b>Batch No.</b>
1-Al	Gd <sub>1.8</sub> Al <sub>0.2</sub> Ti <sub>1.9</sub> O <sub>6.8</sub>	L990602
2-Al	Ca <sub>0.2</sub> Hf <sub>0.2</sub> Gd <sub>1.4</sub> Al <sub>0.2</sub> Ti <sub>1.9</sub> O <sub>6.8</sub>	L990603
3-Al	Ca <sub>0.4</sub> Hf <sub>0.4</sub> Gd <sub>1.0</sub> Al <sub>0.2</sub> Ti <sub>1.9</sub> O <sub>6.8</sub>	L990604
4-Al	Ca <sub>0.6</sub> Hf <sub>0.6</sub> Gd <sub>0.6</sub> Al <sub>0.2</sub> Ti <sub>1.9</sub> O <sub>6.8</sub>	L990605
5-Al	Ca <sub>0.8</sub> Hf <sub>0.8</sub> Gd <sub>0.2</sub> Al <sub>0.2</sub> Ti <sub>1.9</sub> O <sub>6.8</sub>	L990606
1-Mg	Gd <sub>1.8</sub> Mg <sub>0.1</sub> Ti <sub>2.0</sub> O <sub>6.8</sub>	L990607
2-Mg	Ca <sub>0.2</sub> Hf <sub>0.2</sub> Gd <sub>1.4</sub> Mg <sub>0.1</sub> Ti <sub>2.0</sub> O <sub>6.8</sub>	L990608
3-Mg	Ca <sub>0.4</sub> Hf <sub>0.4</sub> Gd <sub>1.0</sub> Mg <sub>0.1</sub> Ti <sub>2.0</sub> O <sub>6.8</sub>	L990609
4-Mg	Ca <sub>0.6</sub> Hf <sub>0.6</sub> Gd <sub>0.6</sub> Mg <sub>0.1</sub> Ti <sub>2.0</sub> O <sub>6.8</sub>	L990610
5-Mg	Ca <sub>0.8</sub> Hf <sub>0.8</sub> Gd <sub>0.2</sub> Mg <sub>0.1</sub> Ti <sub>2.0</sub> O <sub>6.8</sub>	L990611

**Table 3: Sintering conditions for Al- and Mg-containing samples.**

<b>Comp. ID</b>	<b>Temp., °C</b>	<b>Sample ID*</b>
1-Al	1400	#57
2-Al	1400	#58
3-Al	1400	#59
4-Al	1400	#60
5-Al	1400	#61
1-Al	1300	#69
3-Al	1300	#70
5-Al	1300	#71
2-Al	1350	#75
4-Al	1350	#74
1-Mg	1400	#62
2-Mg	1400	#63
3-Mg	1400	#64
4-Mg	1400	#65
5-Mg	1400	#66
1-Mg	1300	#90
3-Mg	1300	#91
5-Mg	1300	#92
2-Mg	1350	#93
4-Mg	1350	#94

\* Recorded sample nos. are L9906--; only the last two digits have been used.

#### 4 RESULTS AND DISCUSSION

The XRD patterns, SEM micrographs and EDS data are listed in Appendices (A) 1-3 respectively for all the sintered samples.

#### 4.1 Al-containing Series

The XRD patterns for the five compositions listed in Table 2, sintered at 1300<sup>0</sup>-1400<sup>0</sup>C for 16 h, are shown in A1, Figs. 1-10. Their SEM micrographs are shown in A2, Figs. 1-10 and the EDS analyses of the phases observed are listed in A3, Tables 1-9. The phases observed for the ten samples are summarised in Table 4. The phases observed are the same at the different temperatures sintered for the five compositions. There were slight differences between the phases observed by SEM and XRD. The presence of Al has suppressed the formation of 4M zirconolite, which was observed in the pseudo-binary system CaHfTi<sub>2</sub>O<sub>7</sub>-Gd<sub>2</sub>Ti<sub>2</sub>O<sub>7</sub>[1]. Pyrochlore is present only in compositions 1-Al to 3-Al only at all sintering temperatures. Most of the Al is present in both pyrochlore and zirconolite. Hf in the Gd site provides charge compensation for pyrochlore-rich compositions. Gd in the Ca site provides charge compensation zirconolite-rich compositions; Al entering Ti site in both cases. However, a trace of aluminium titanate was present in composition 1-Al and a trace of alumina in 2-Al at 1400<sup>0</sup>C (Table 4). Perovskite was present in composition 4-Al. The grain size was < 1 µm for the 1300<sup>0</sup>C sintering, however it was possible to analyse by EDS all the samples except sample #70 (composition 3-Al). The approximate phase boundaries have been marked on Figure 1, taking into consideration the phases observed previously [1].

**Table 4: Summary of phases observed in the Al-containing Series.**

Comp.ID	Sample ID	Sintering temp., °C	Phases observed	
			SEM/EDS*	XRD*
1-Al	#57	1400	P (M), AT(t)	P (M)
2-Al	#58	1400	P (M), Z (m), A (t)	P (M), Z (m),
3-Al	#59	1400	P (M), Z (M)	P (M), Z (M)
4-Al	#60	1400	Z (M), Pk (m)	Z (M), Pk (m)
5-Al	#61	1400	Z (M), R (m)	Z (M)
1-Al	#69	1300	P (M), G (m)	P (M),
3-Al	#70	1300	P (M), Z (M)	P (M), Z (M)
5-Al	#71	1300	Z (M), R (m)	Z (M)
2-Al	#75	1350	P (M), Z (m)	P (M), Z (m)
4-Al	#74	1350	Z (M), P (m)	Z (M), Pk (m)

\* Key: M= major phase; m= minor phase; t= trace; P= pyrochlore; Z= 2M zirconolite; Pk= perovskite; AT= Al<sub>2</sub>TiO<sub>5</sub>; A= Al<sub>2</sub>O<sub>3</sub>; R= Hf-rutile; G= Gd/Al titanate.

#### 4.2 Mg-containing Series

The XRD patterns for the five compositions listed in Table 2, sintered at 1300<sup>0</sup>-1400<sup>0</sup>C for 16 h, are shown in A1, Figs. 11-21. Their SEM micrographs are shown in A2, Figs. 12-22 and the EDS analyses of the phases observed are listed in A3, Tables 10-20. The phases observed for the ten samples are summarised in Table 5. The phases observed at 1400<sup>0</sup>C and below are similar except for a trace of zirconolite found in sample #93 sintered at 1350<sup>0</sup>C. The rutile observed by SEM was evidently too small to be seen in the XRD traces. In contrast to the Al-containing materials, rutile was present in all the Mg-

containing samples. The rutile contained Hf, except for composition 1-Mg. Mg was present in both pyrochlore and zirconolite. To distinguish between the pyrochlore and

**Table 5: Summary of phases observed in the Mg-containing Series.**

Comp.ID	Sample ID	Sintering temp., °C	Phases observed	SEM/EDS*	XRD*
1-Mg	#62	1400		P (M), R(t)	P (M)
2-Mg	#63	1400		P (M), H (m)	P (M)
3-Mg	#64	1400		Z (M), H (m)	Z (M)
4-Mg	#65	1400		P (m), Z (M), H (m)	Z (M)
5-Mg	#66	1400		Z (M), H (m)	Z (M)**
1-Mg	#90	1300		P (M), R (m)	P (M)
3-Mg	#91	1300		Z (M), H (m)	Z (M)
5-Mg	#92	1300		Z (M), H (m)	Z (M)
2-Mg	#93	1350		P (M), Z (m)	P (M), Z (t)
4-Mg	#94	1350		P (m), Z (M), H (m)	Z (M)**

\*Key: M= major phase; m= minor phase; t= trace; P= pyrochlore; Z= 2M zirconolite; Pk= perovskite; R= rutile; H= Hf-rutile.

\*\* Trace of 4M zirconolite.

zirconolite in the SEM was more difficult than for the Al-containing samples, as seen in A2 Figs. 14/15 and 21/22, where the contrast had to be increased. In composition 4-Mg at 1400°C (#65) and 1300°C (#94) two phases have been identified in addition to rutile by SEM (Figs. 15 and 22). These have been designated as pyrochlore and zirconolite (Tables 13 and 19). However, the XRD traces (Figs. 14 and 20) do not show the presence of pyrochlore but rather a trace of 4M zirconolite. It is possible that the phase designated as pyrochlore in the EDS analysis could be a 4M zirconolite. Mg is likely to be at the Hf site which pushes Gd into the Ca site and this would likely to result in 4M zirconolite. It is clearly seen that the presence of Mg suppresses the formation of 4M zirconolite. The approximate phase boundaries are marked in Figure 2.

#### 4.3 General Comments

Al and Mg systems have approximately same effects as the binary pyrochlore-zirconolite system. Perovskite was present in the Al-containing system but was not observed in the Mg-containing system. In the Mg-containing system the phase region of pyrochlore and zirconolite is much smaller than in the Al-containing system. To ascertain the extent of the phase boundaries more compositions are being prepared.

#### 5 References

- 1 M. W. A. Stewart, E. R. Vance and R. A. Day, Supplement to Interim Report on Task 1.3: Equilibrium Phase Diagram. ANSTO Report R99m023, 18 April 99.

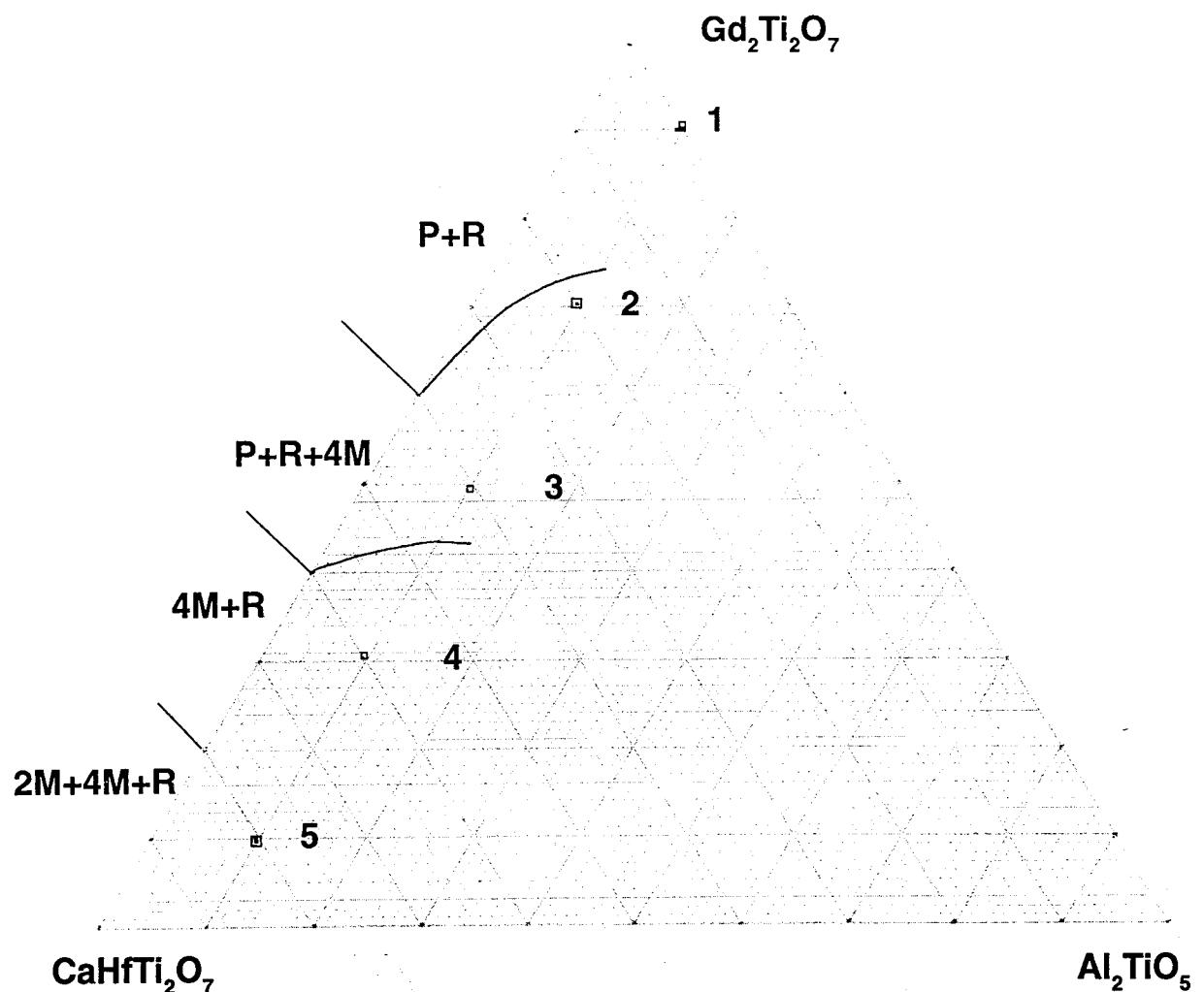


Figure 1. Pseudo-ternary phase equilibrium diagram at 1400°C showing the approximate phase boundaries for the compositions (1-5) investigated.

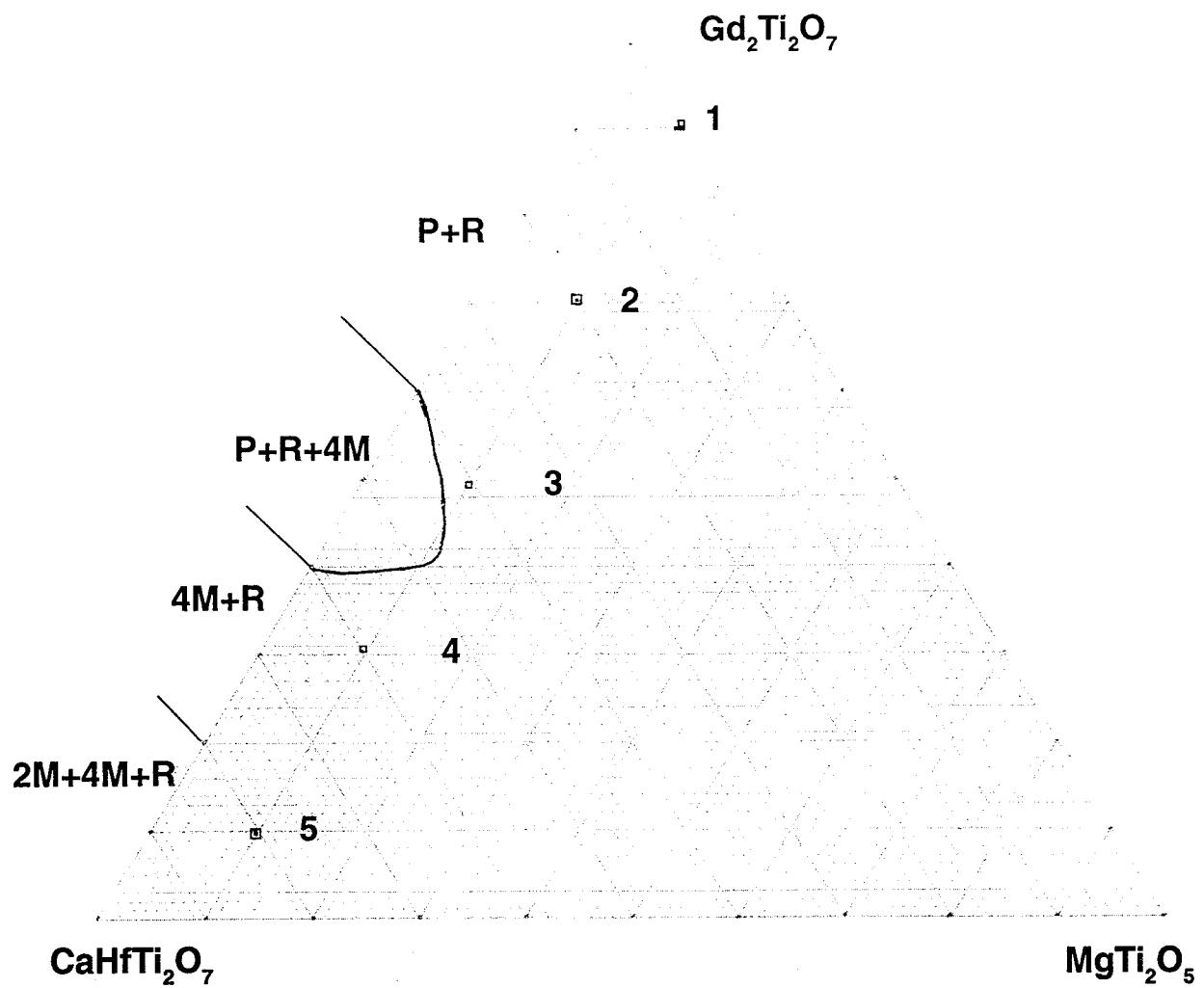


Figure 2. Pseudo-ternary phase equilibrium diagram at 1400°C showing the approximate phase boundaries for the compositions (1-5) investigated.

## **APPENDICES**

### **Appendix 1**

#### **XRD Patterns**

Figures 1- 20

### **Appendix 2**

#### **SEM Micrographs**

Figures 1- 22

### **Appendix 3**

#### **EDS Analysis**

Tables 1-19

## **Appendix 1**

### **XRD Patterns**

Figures 1- 20

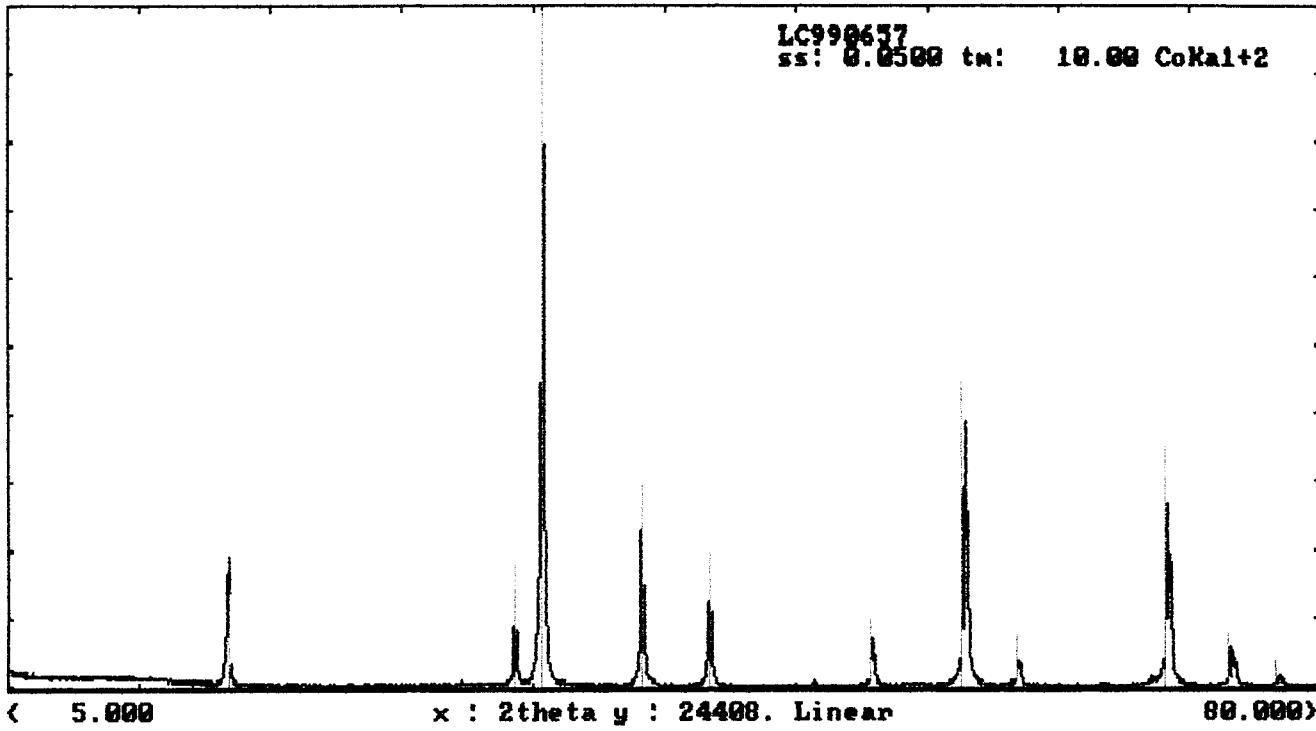


Figure 1. XRD pattern for sample #57 (1-Al) sintered at 1400<sup>0</sup>C showing pyrochlore as the major phase.

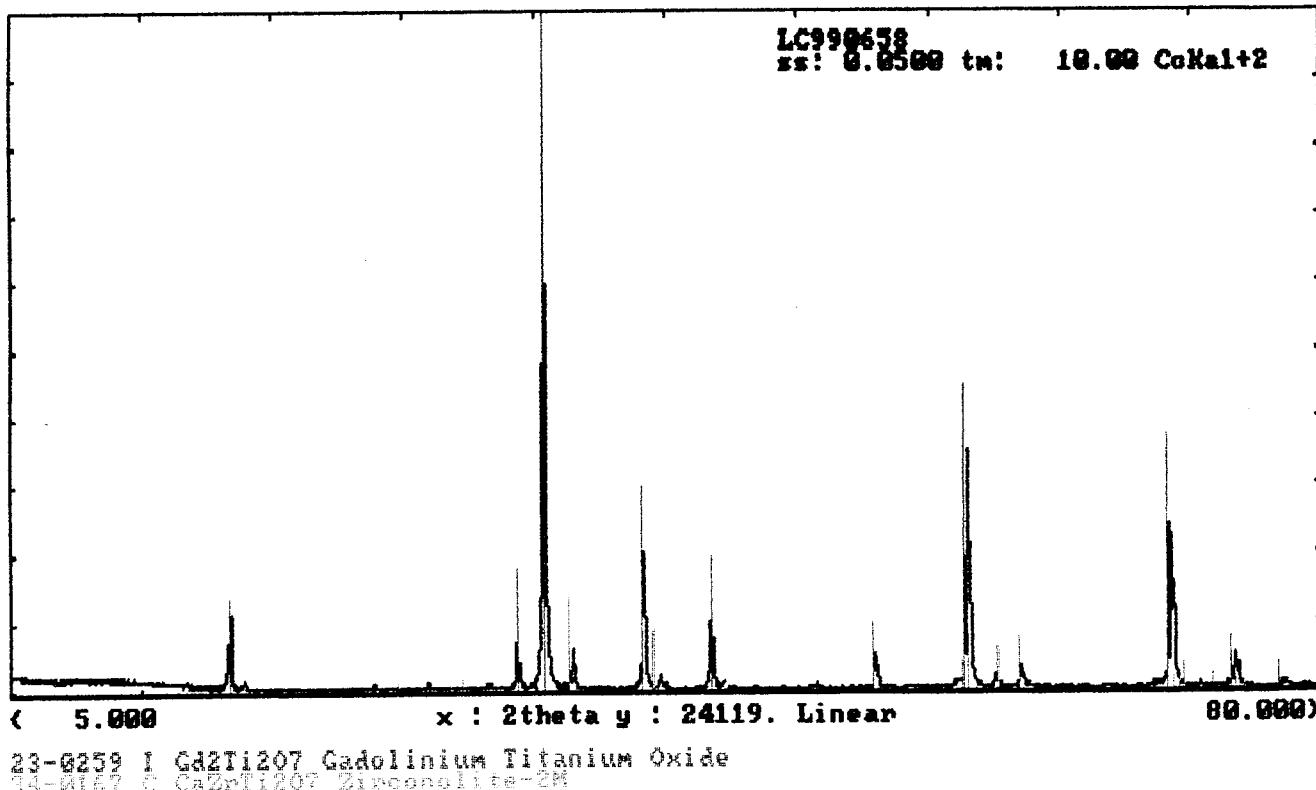


Figure 2. XRD pattern for sample #58 (2-Al) sintered at 1400<sup>0</sup>C showing pyrochlore (major phase) and 2M zirconolite (minor phase).

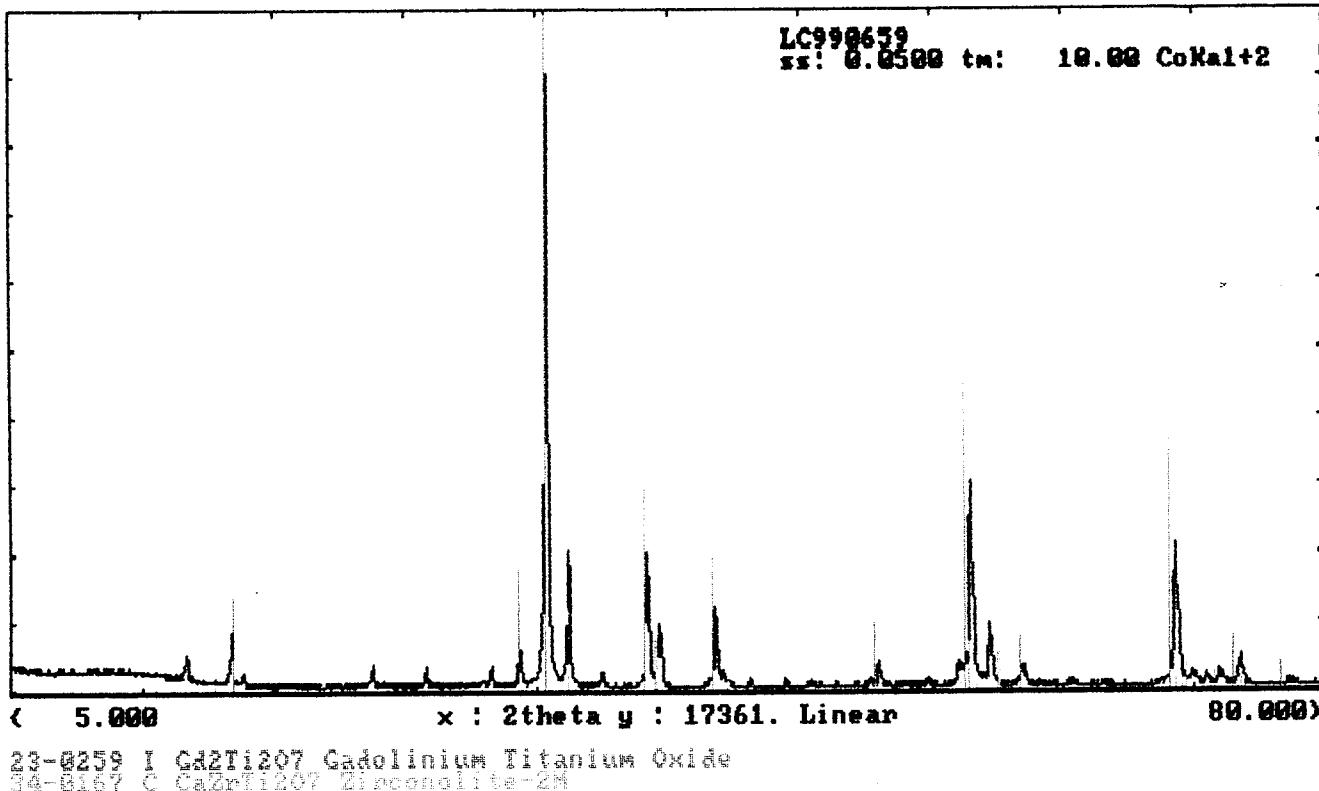


Figure 3. XRD pattern for sample #59 (3-Al) sintered at 1400°C showing pyrochlore and 2M zirconolite.

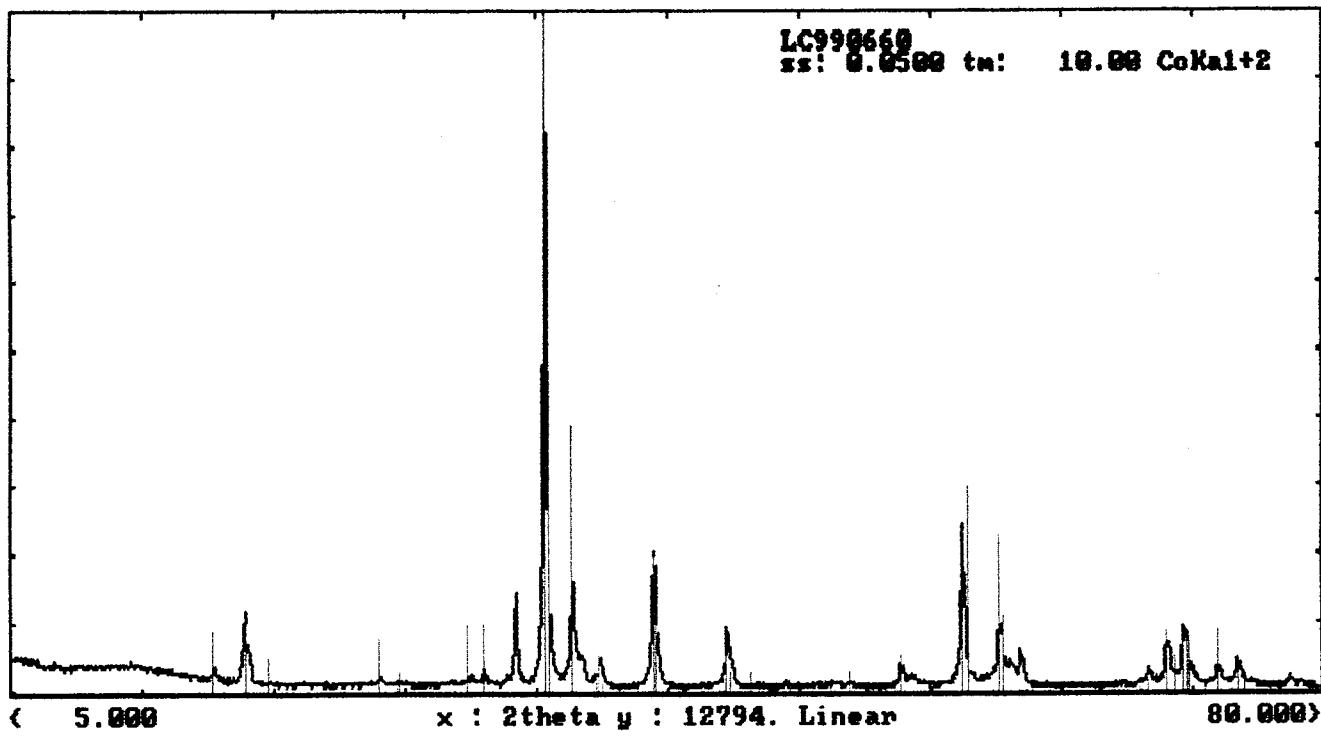


Figure 4. XRD pattern for sample #60 (4-Al) sintered at 1400°C showing perovskite (minor phase) and 2M zirconolite (major phase).

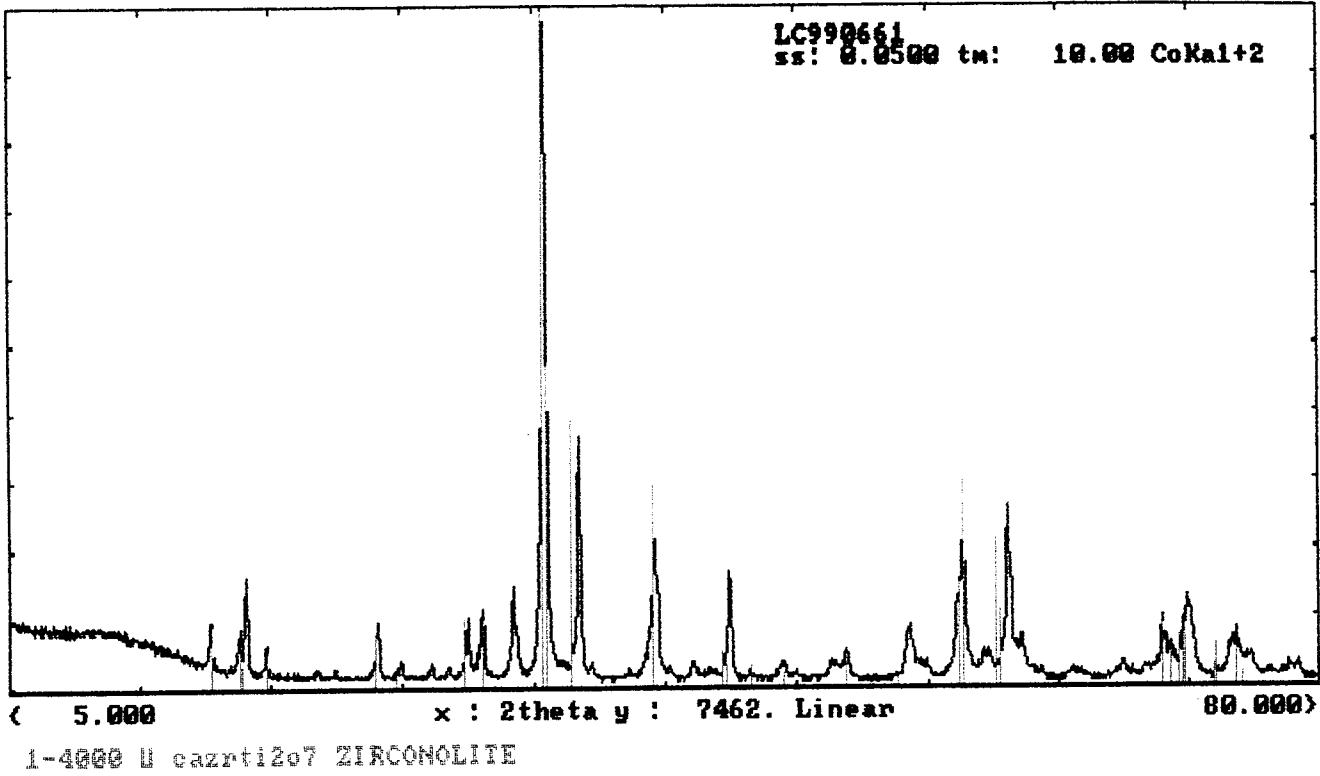


Figure 5. XRD pattern for sample #61 (5-Al) sintered at 1400°C showing 2M zirconolite as the major phase.

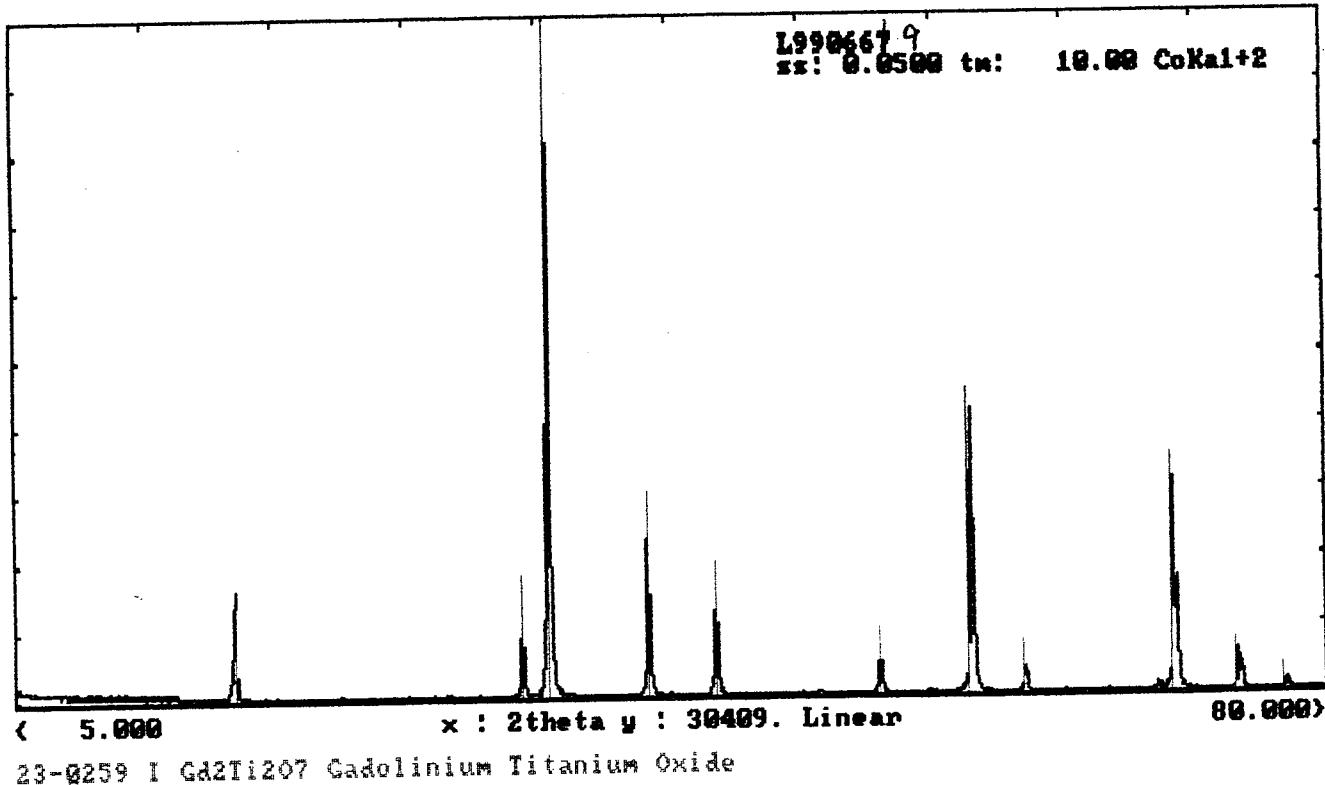


Figure 6. XRD pattern for sample #69 (1-Al) sintered at 1300°C showing pyrochlore as the major phase.

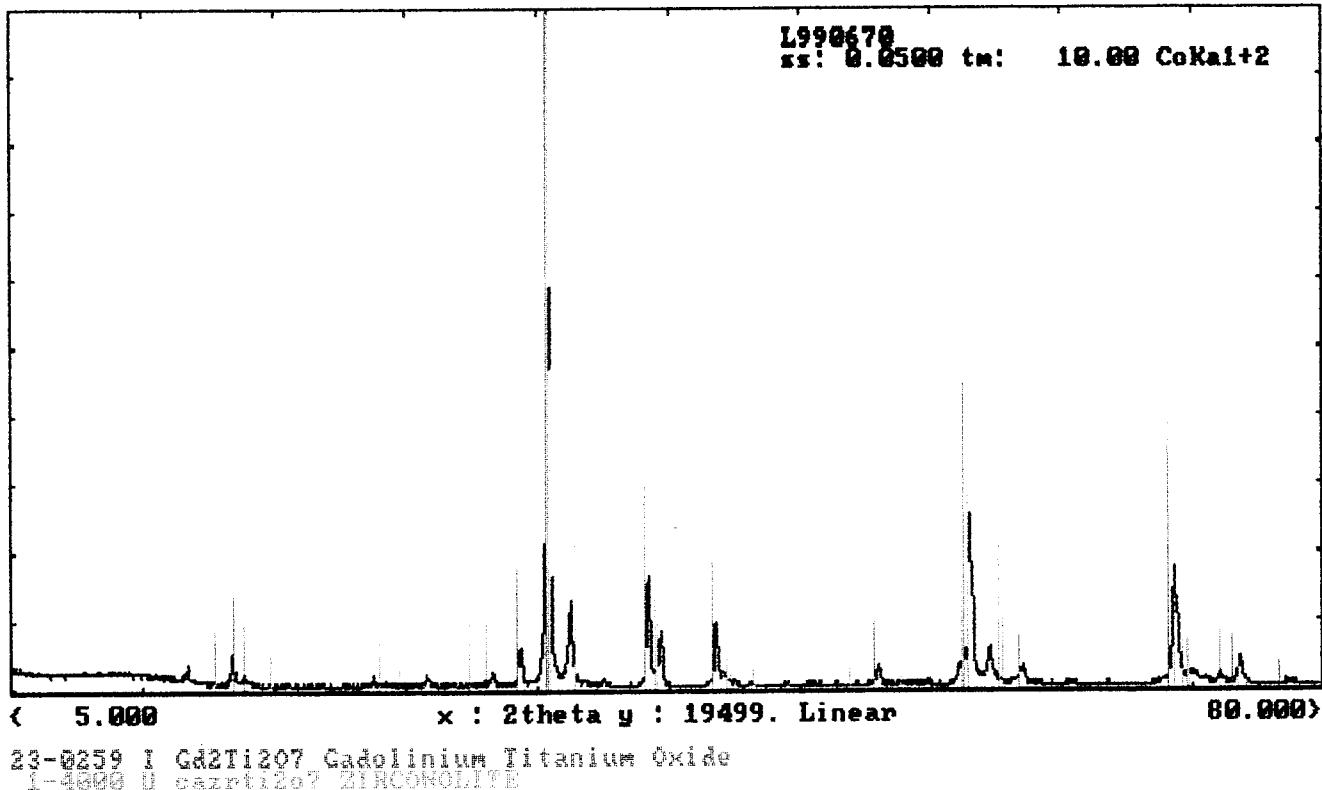


Figure 7. XRD pattern for sample #70 (3-Al) sintered at 1300°C showing pyrochlore and 2M zirconolite.

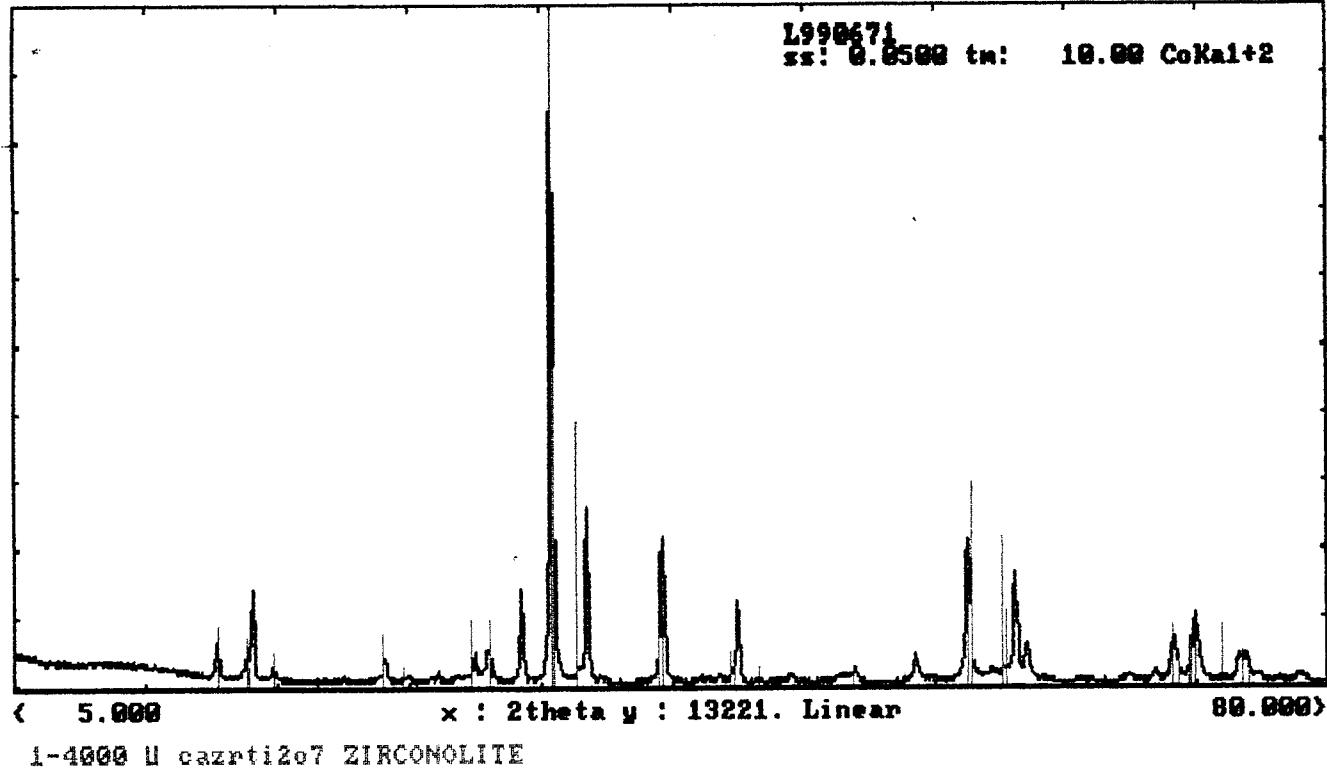


Figure 8. XRD pattern for sample #71 (5-Al) sintered at 1300°C showing 2M zirconolite as the major phase.

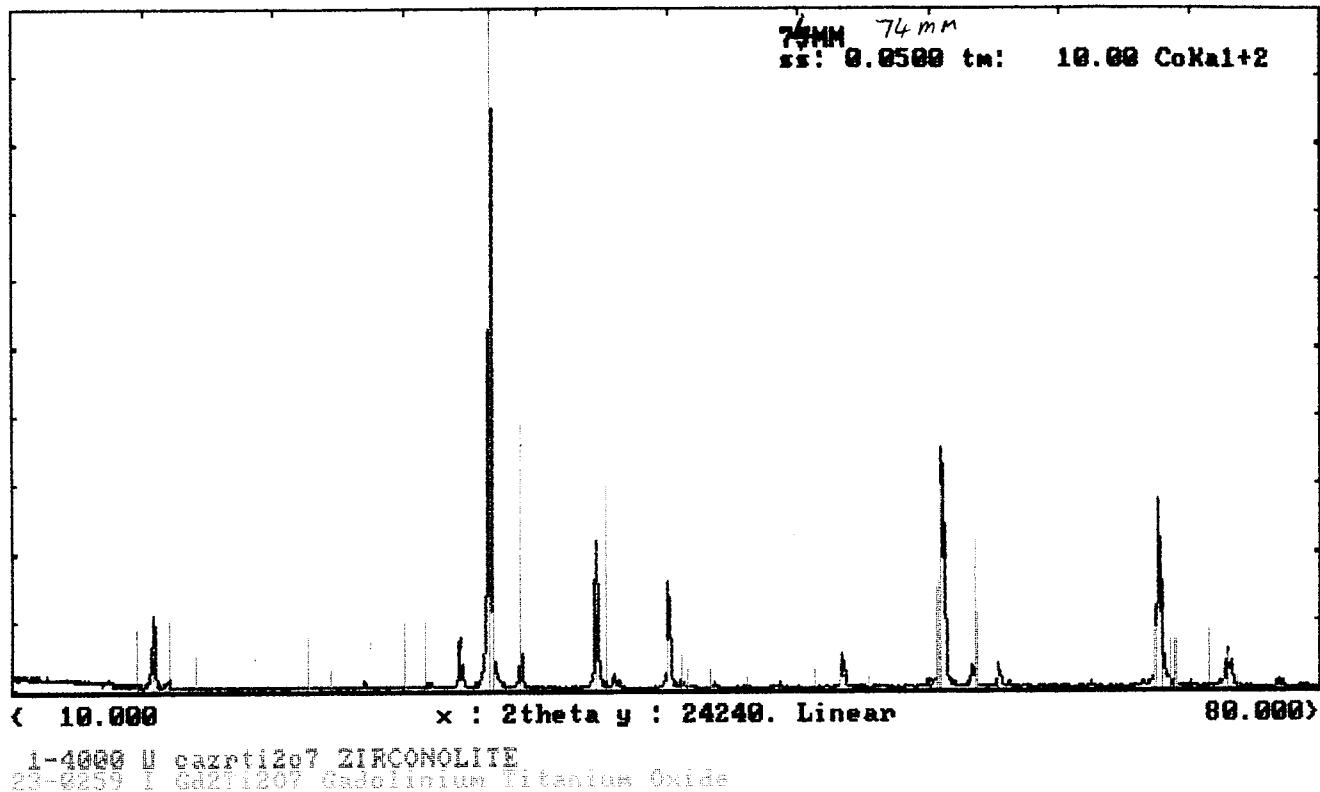


Figure 9. XRD pattern for sample #74 (2-Al) sintered at 1350°C showing pyrochlore and 2M zirconolite.

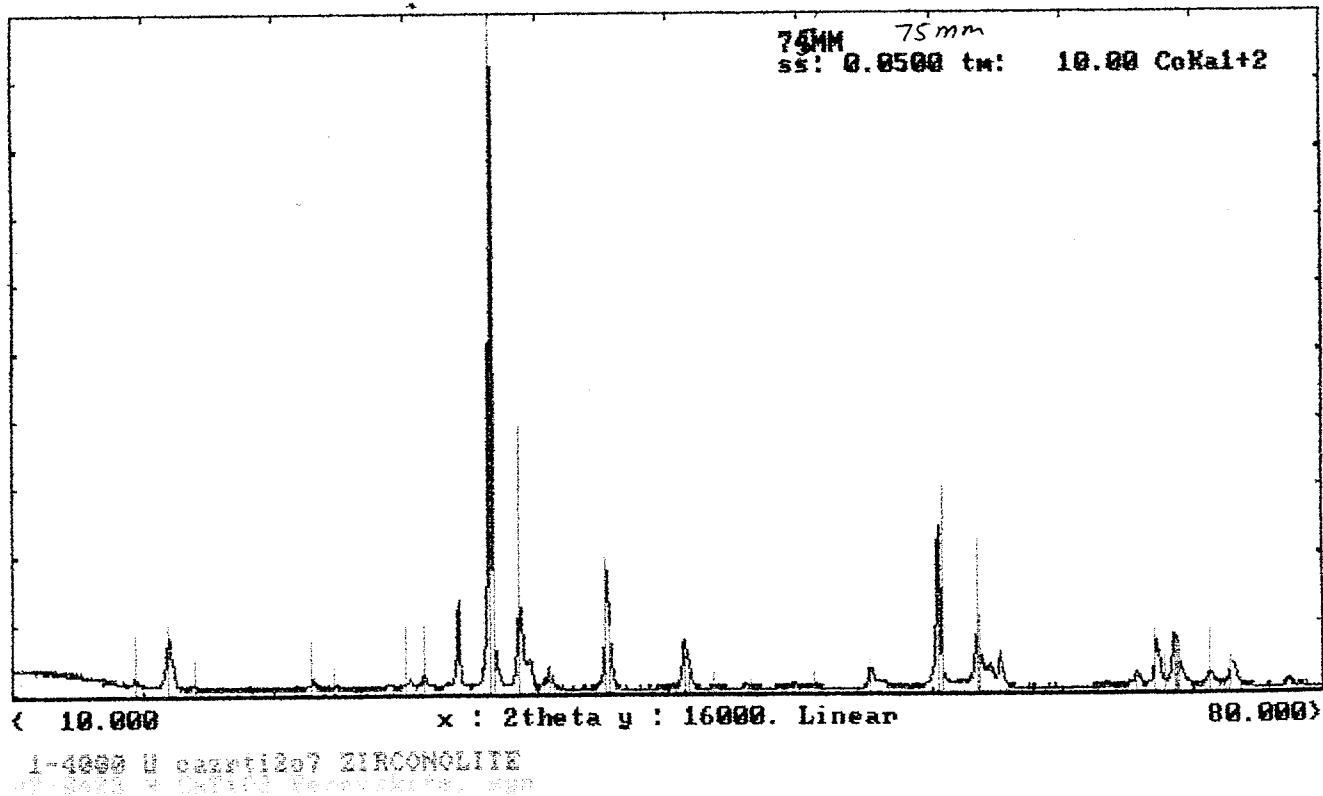
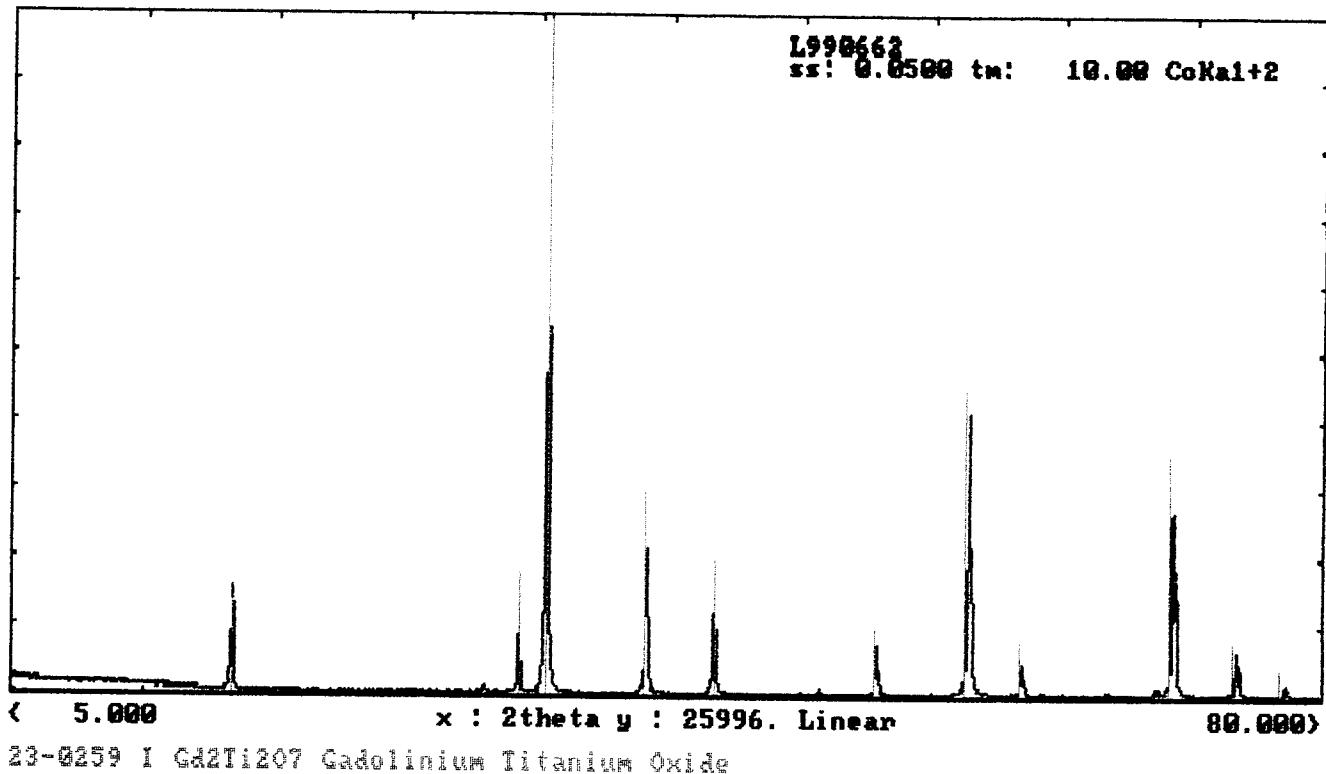
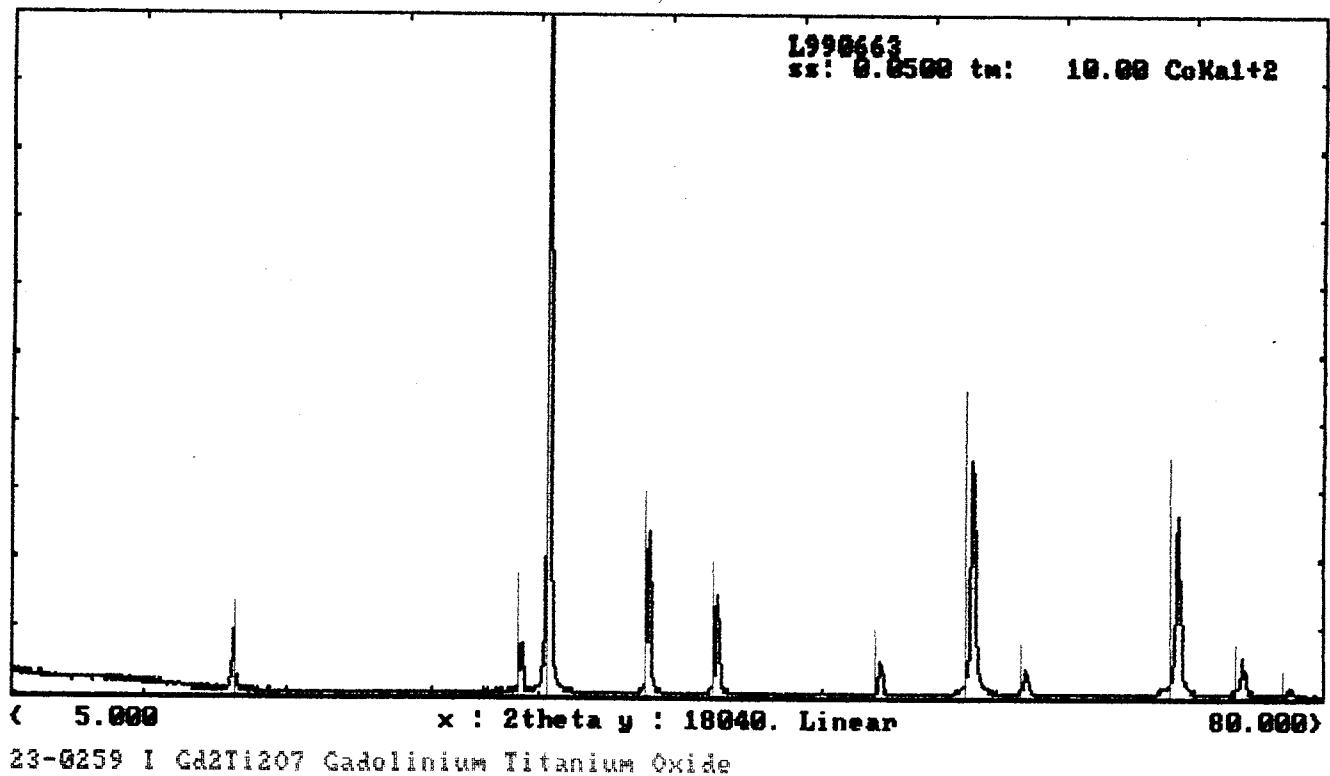


Figure 10. XRD pattern for sample #75 (4-Al) sintered at 1350°C showing perovskite (minor phase) and 2M zirconolite (major phase).



**Figure 11.** XRD pattern for sample #62 (1-Mg) sintered at 1400°C showing pyrochlore as the major phase.



**Figure 12.** XRD pattern for sample #63 (2-Mg) sintered at 1400°C showing pyrochlore as the major phase.

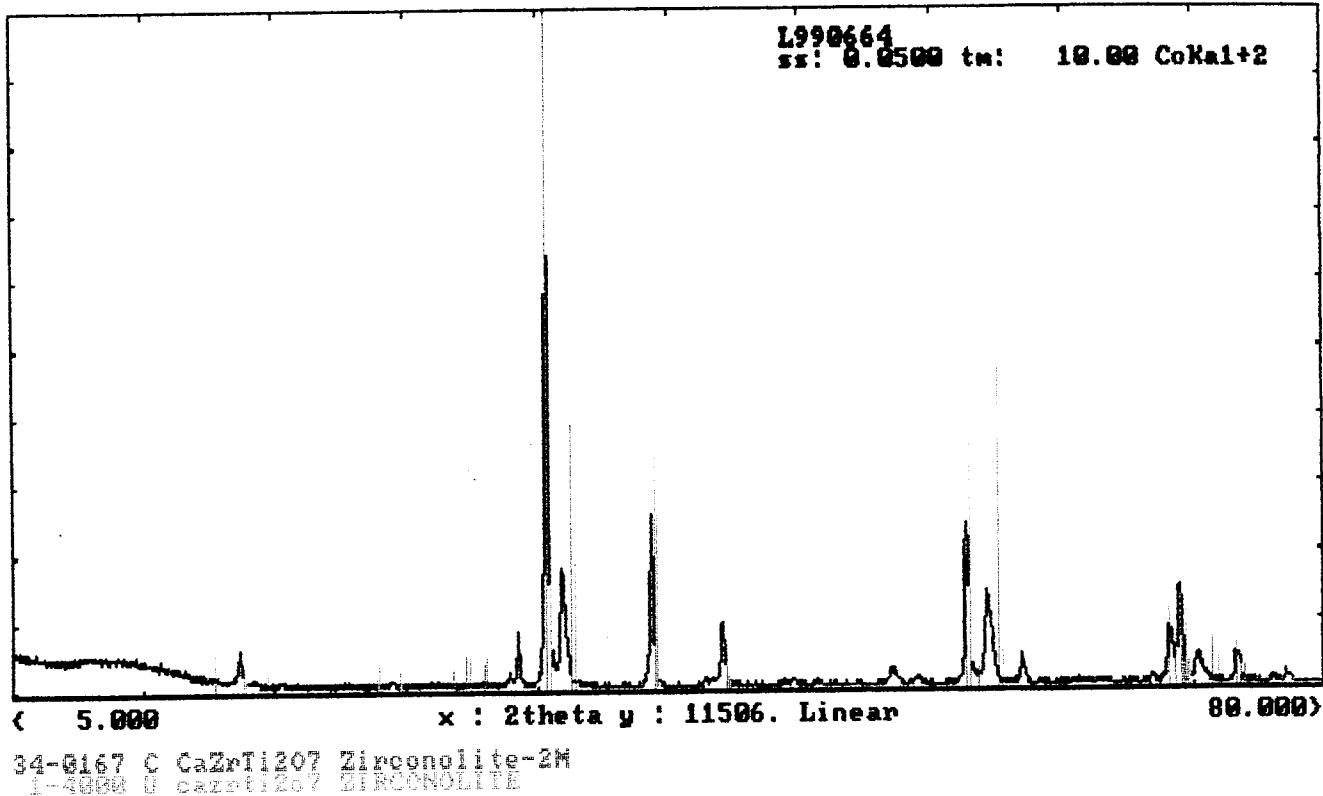


Figure 13. XRD pattern for sample #64 (3-Mg) sintered at 1400°C showing 2M zirconolite as the major phase.

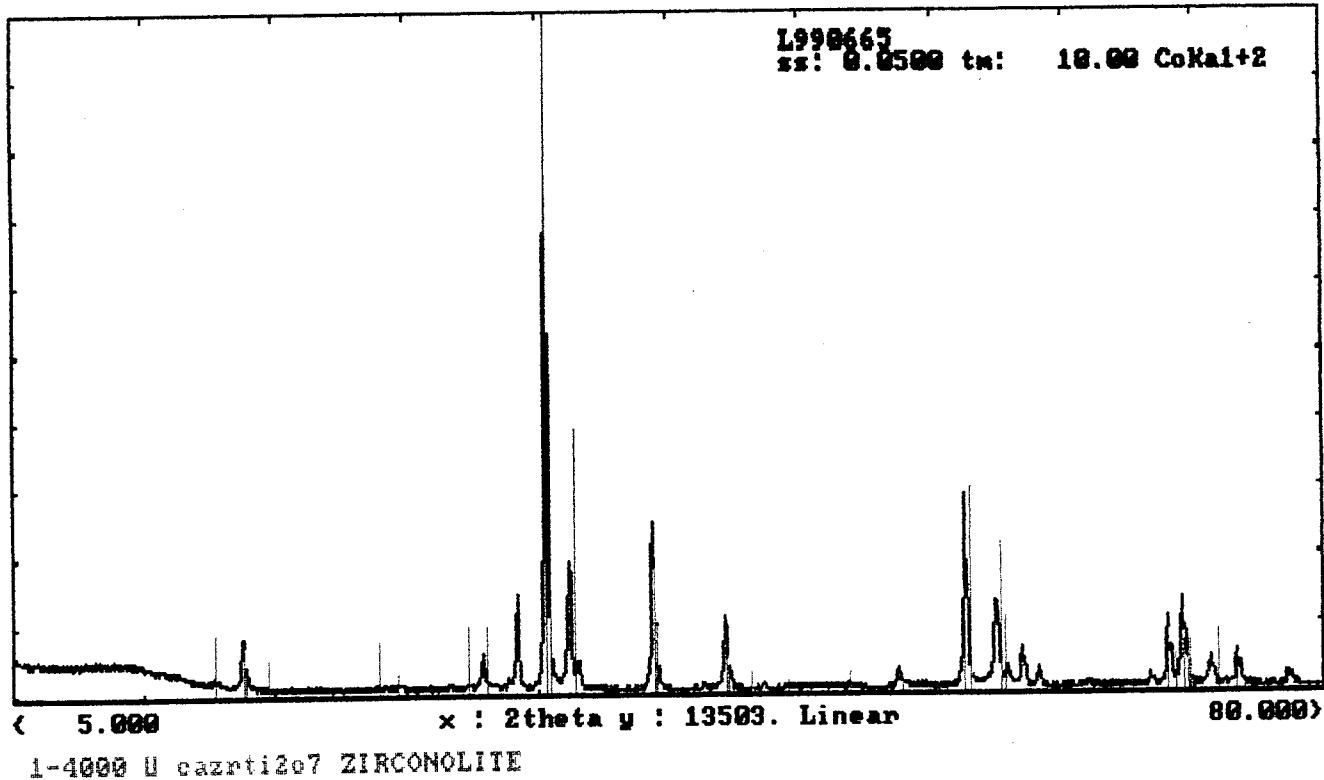


Figure 14. XRD pattern for sample #65 (4-Mg) sintered at 1400°C showing 2M zirconolite as the major phase.

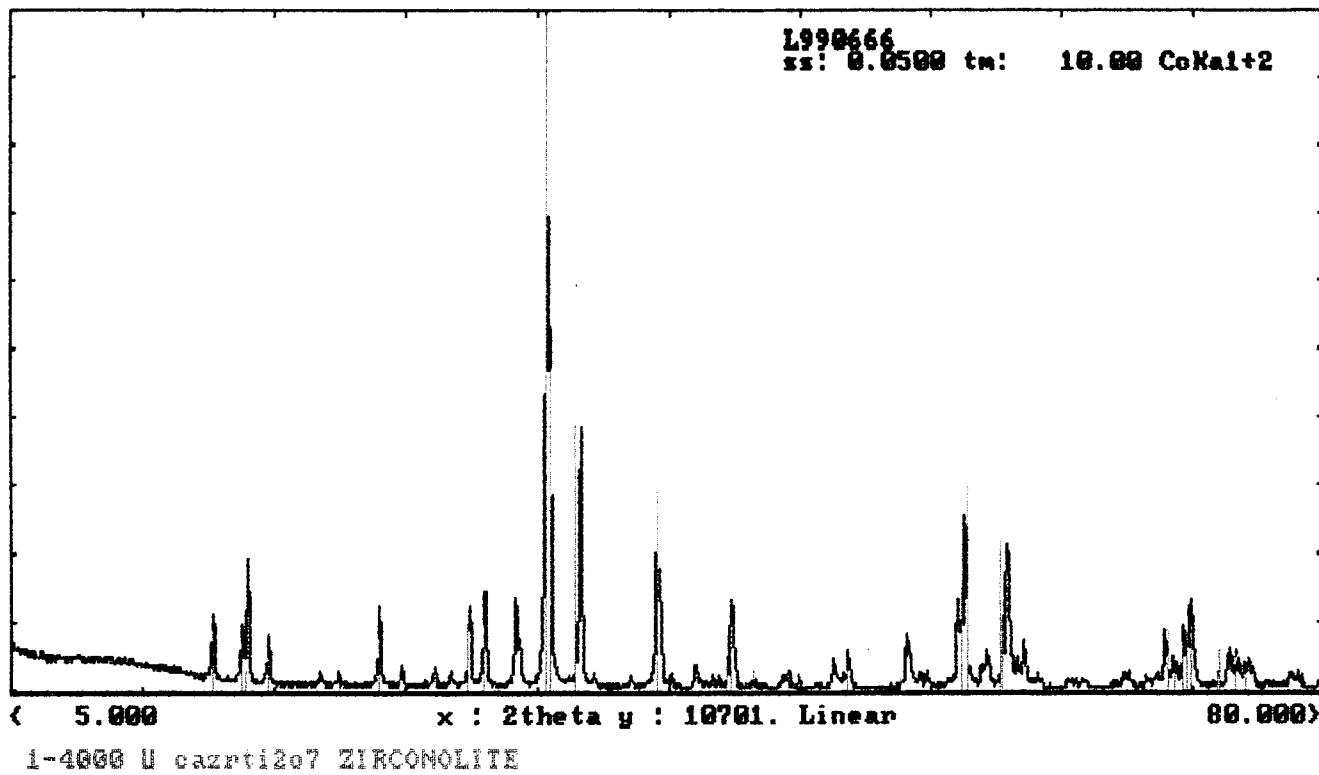


Figure 15. XRD pattern for sample #66 (5-Mg) sintered at 1400°C showing 2M zirconolite as the major phase.

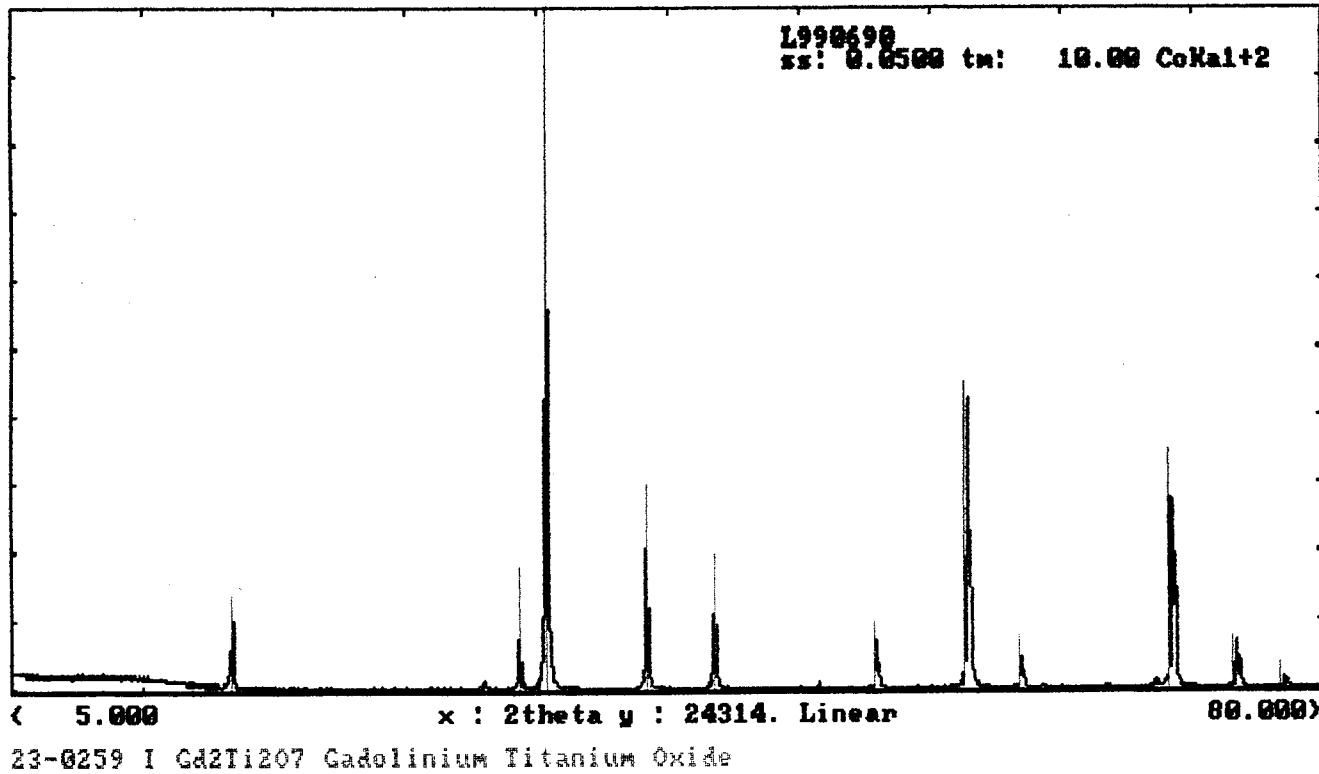


Figure 16. XRD pattern for sample #90 (1-Mg) sintered at 1300°C showing pyrochlore as the major phase.

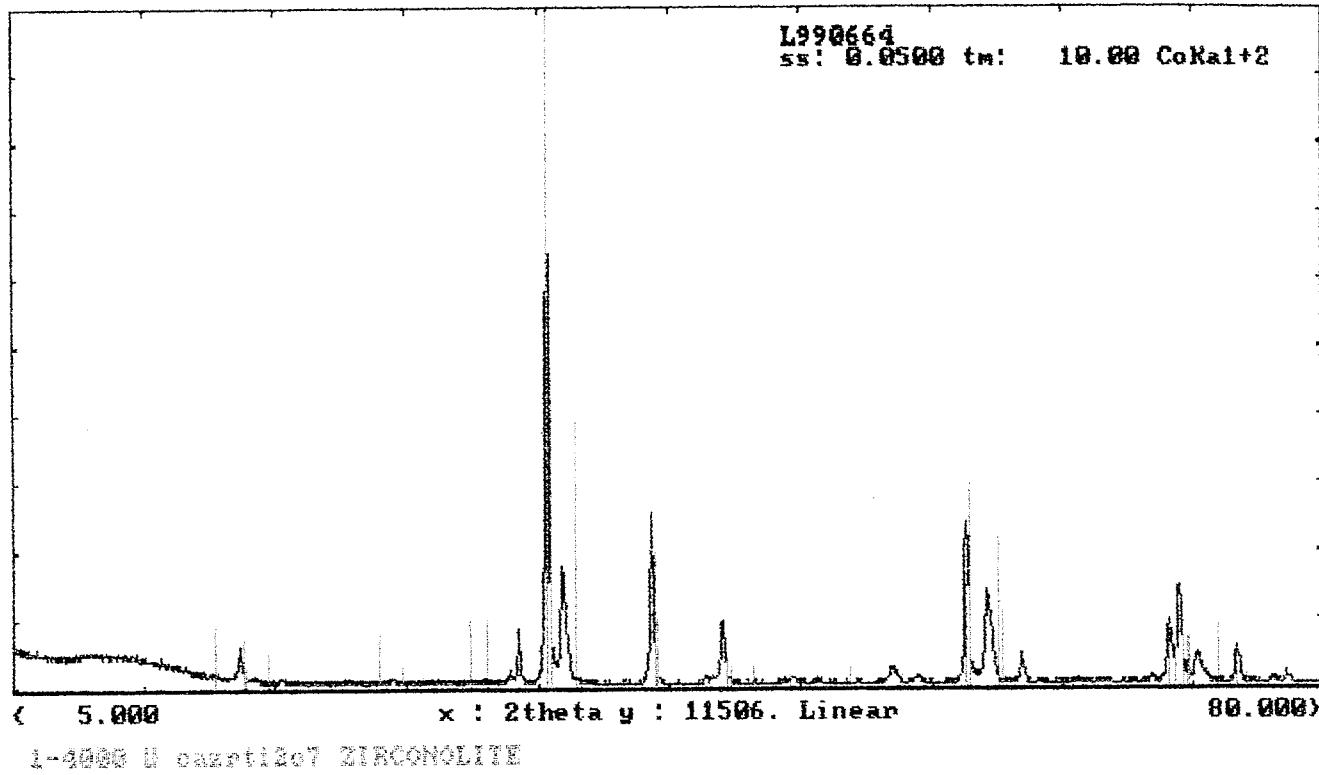


Figure 17. XRD pattern for sample #91 (3-Mg) sintered at 1300°C showing 2M zirconolite as the major phase.

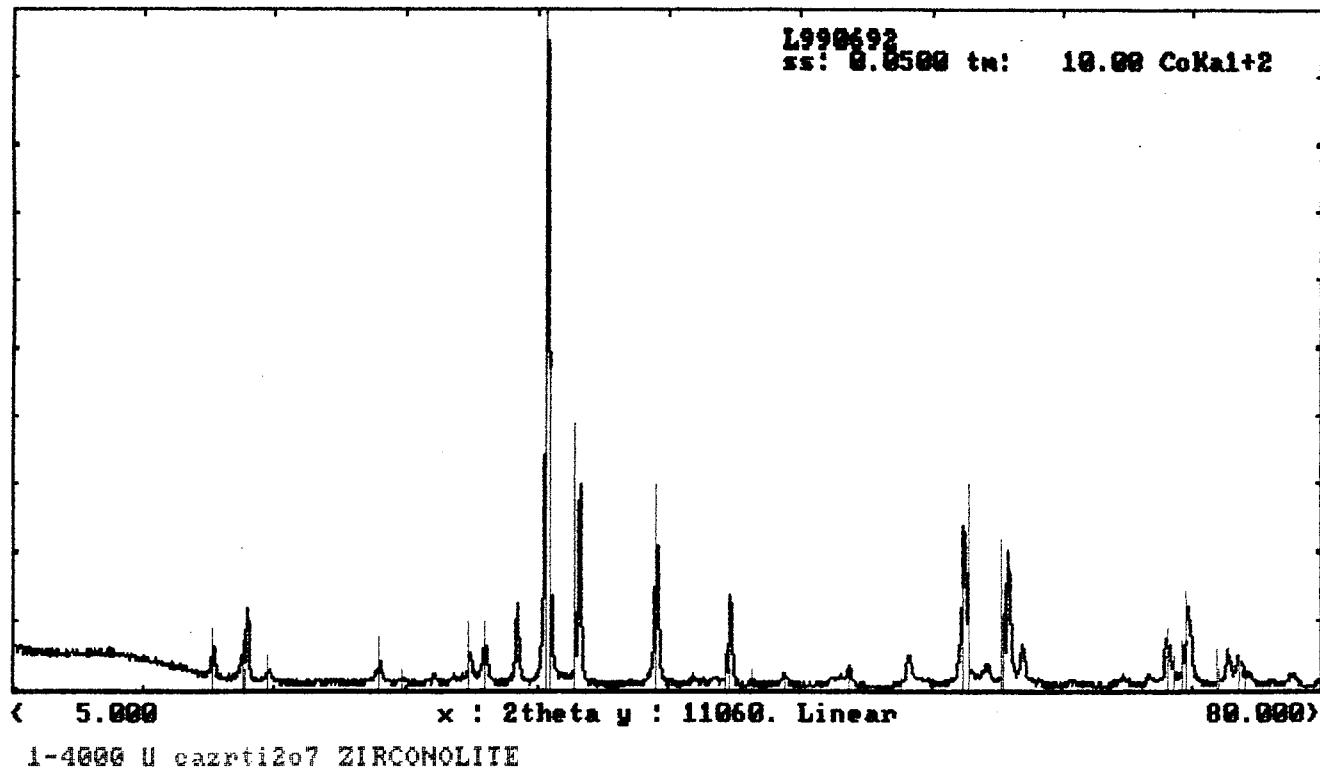
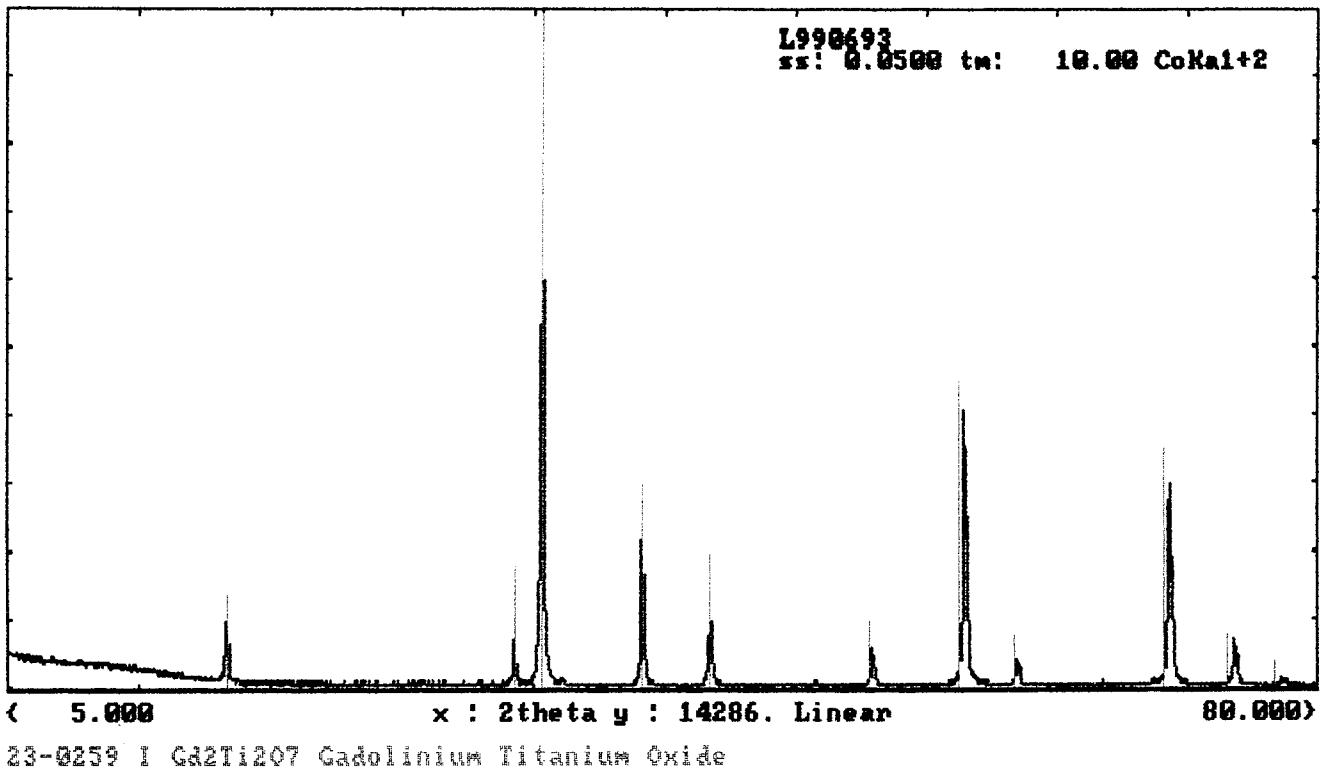
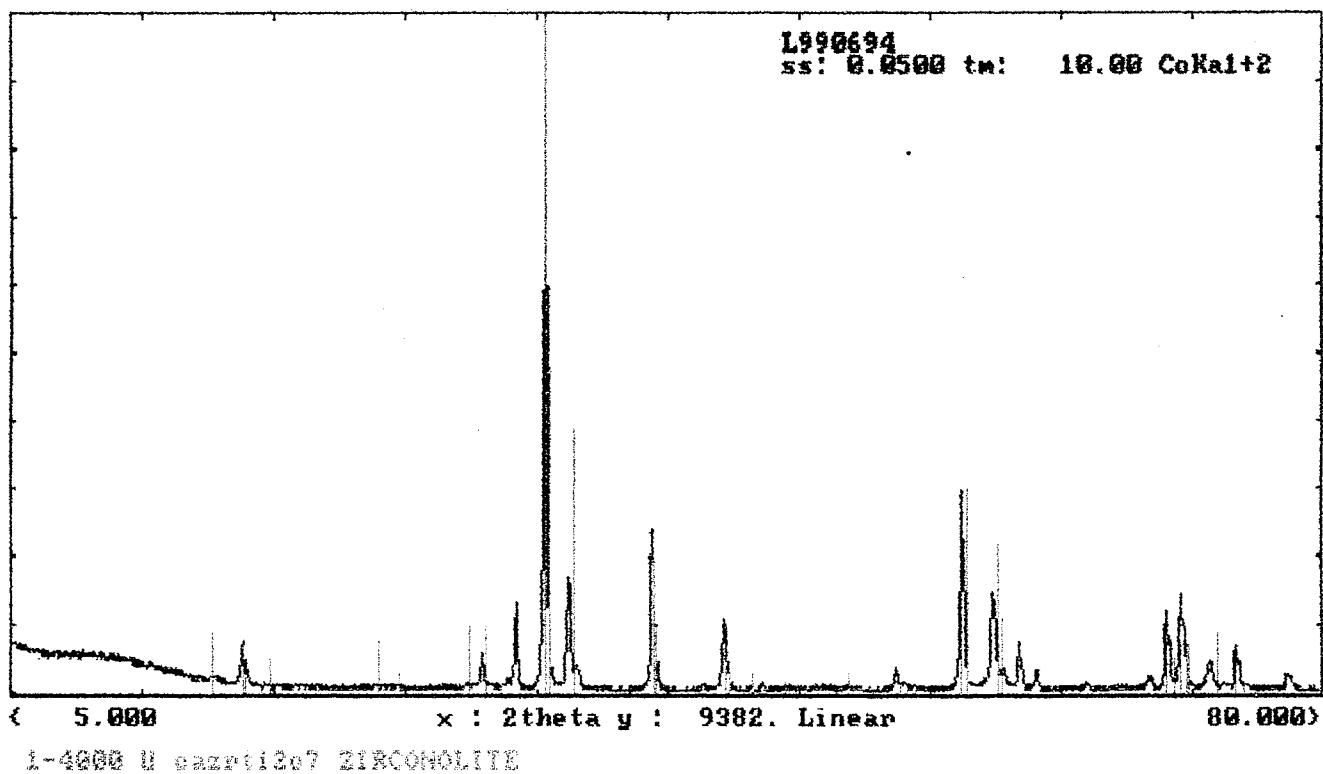


Figure 18. XRD pattern for sample #92 (5-Mg) sintered at 1300°C showing 2M zirconolite as the major phase.



**Figure 19.** XRD pattern for sample #93 (2-Mg) sintered at 1350°C showing pyrochlore as the major phase.



**Figure 20.** XRD pattern for sample #94 (4-Mg) sintered at 1350°C showing 2M zirconolite as the major phase.

## **Appendix 2**

### **SEM Micrographs**

Figures 1- 22

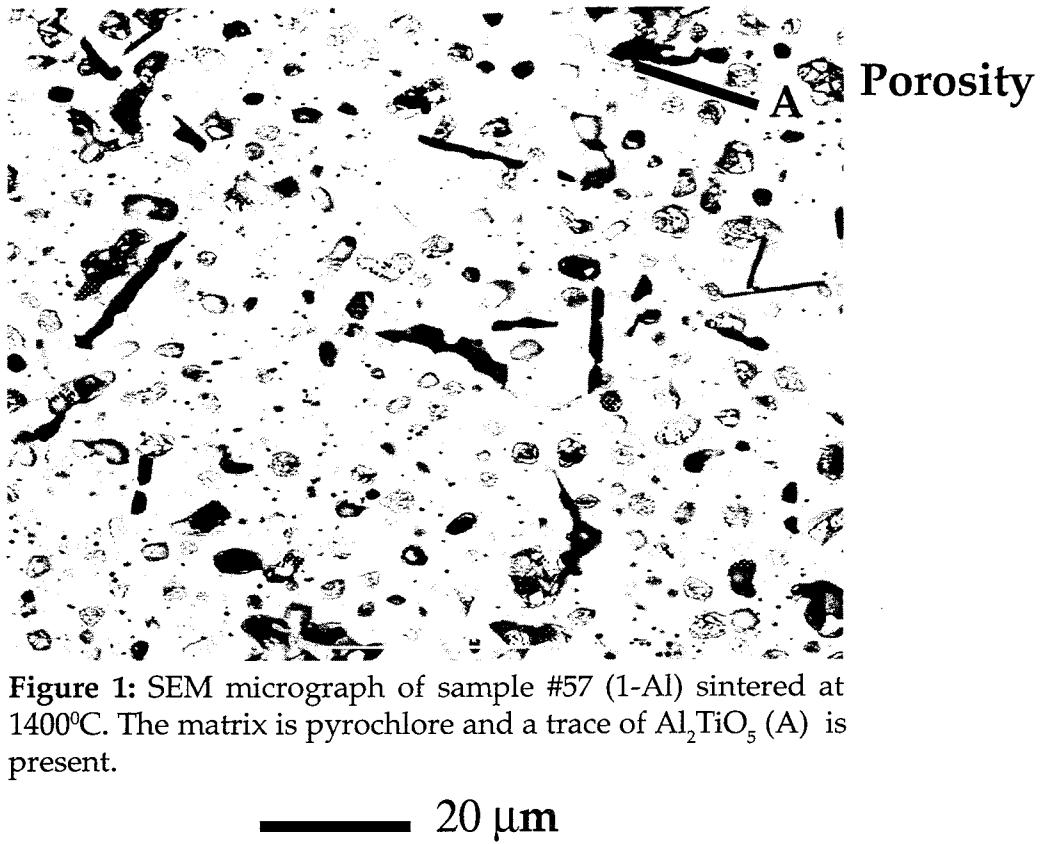


Figure 1: SEM micrograph of sample #57 (1-Al) sintered at 1400°C. The matrix is pyrochlore and a trace of  $\text{Al}_2\text{TiO}_5$  (A) is present.

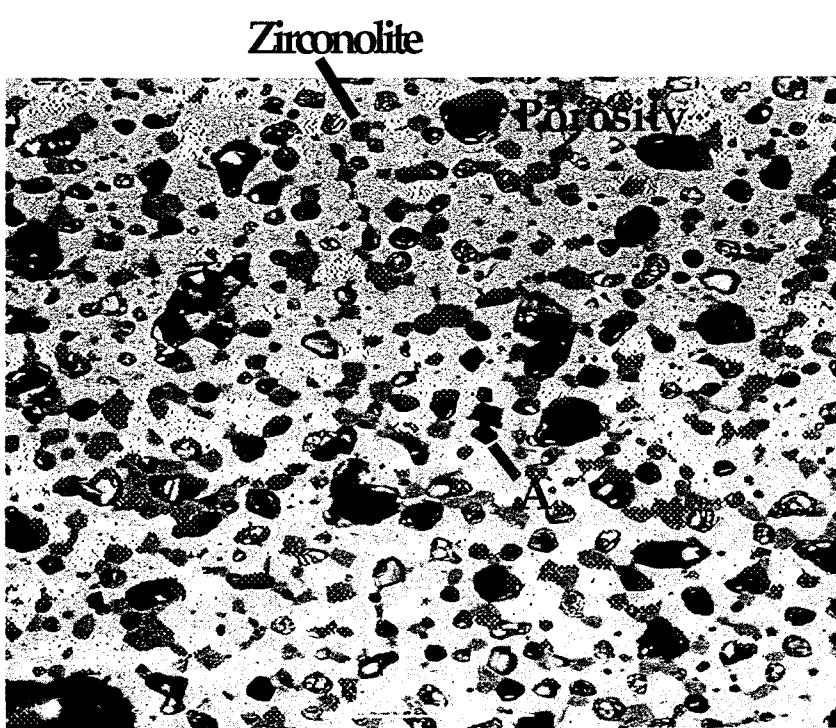


Figure 2: SEM micrograph of sample #58 (2-Al) sintered at 1400°C. The matrix is pyrochlore with 2M zirconolite as a minor phase and a trace of  $\text{Al}_2\text{O}_3$  (A) is present.

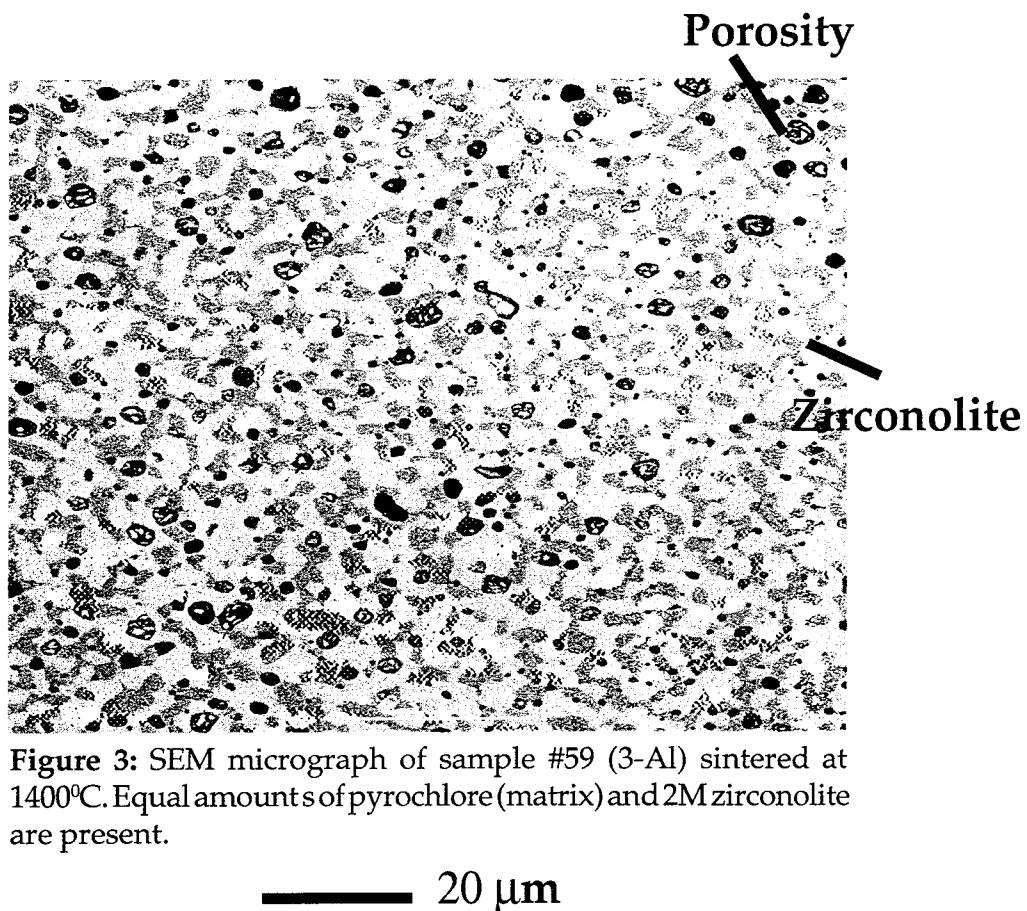


Figure 3: SEM micrograph of sample #59 (3-Al) sintered at 1400°C. Equal amounts of pyrochlore (matrix) and 2M zirconolite are present.

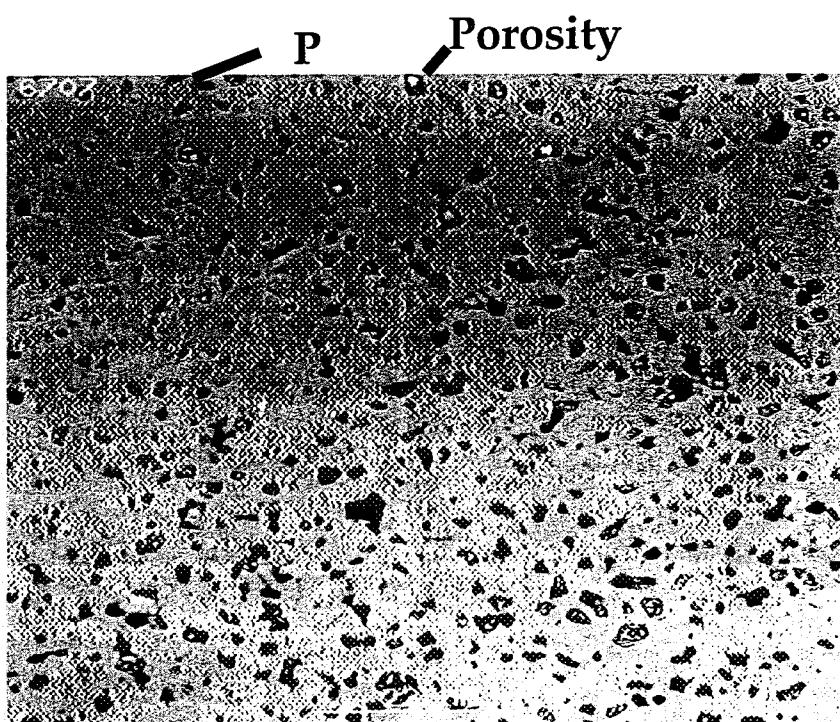
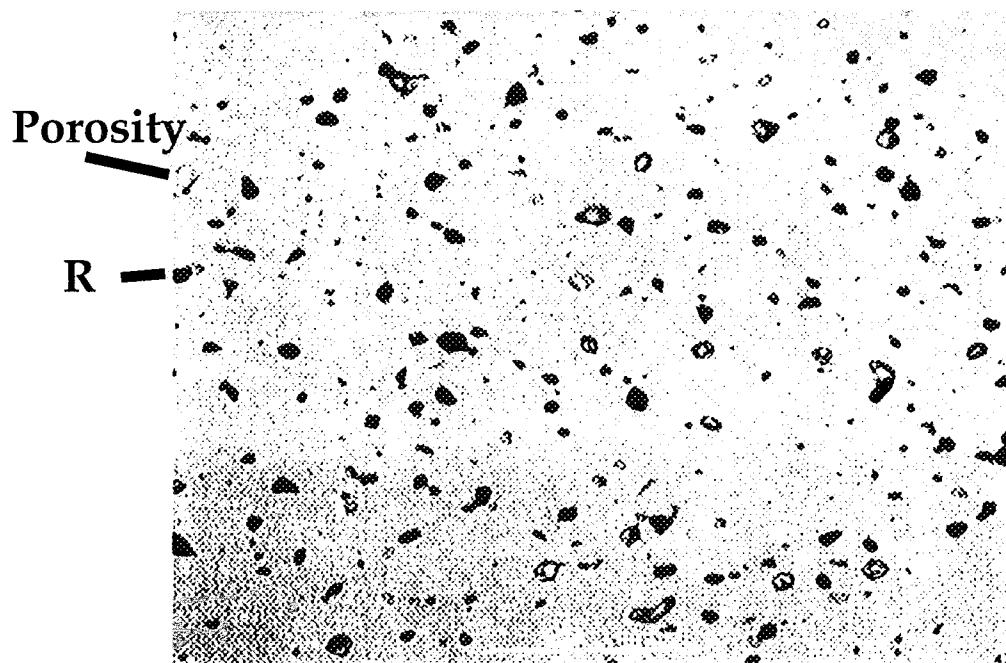
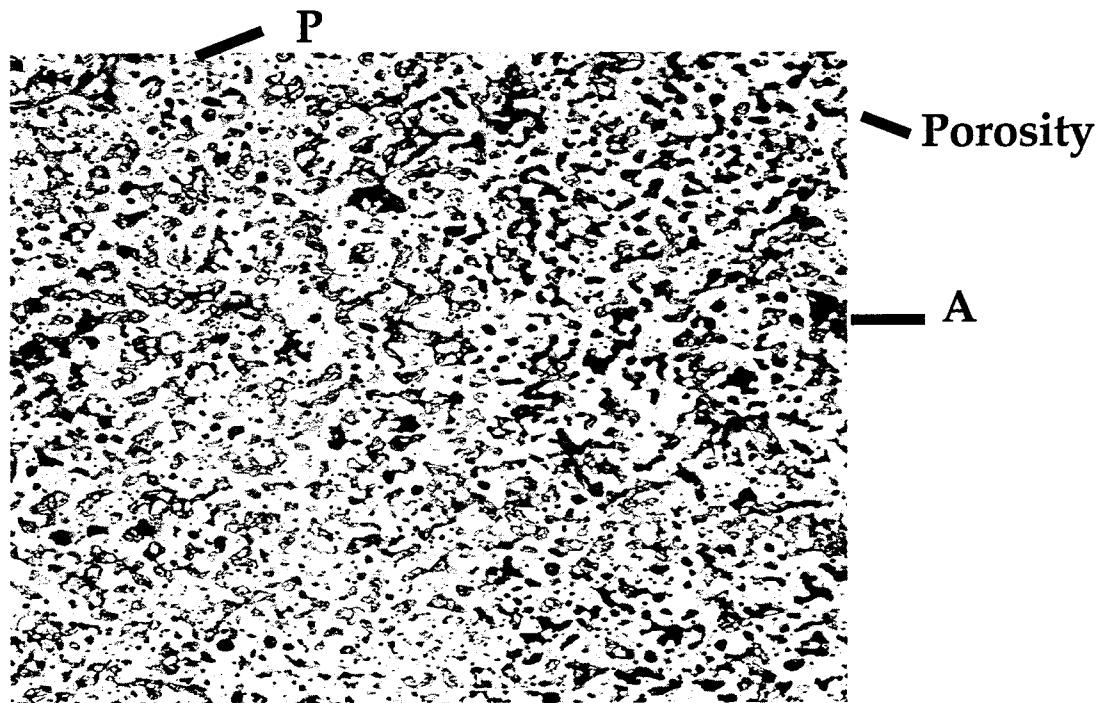


Figure 4: SEM micrograph of sample #60 (4-Al) sintered at 1400°C. The matrix is 2M zirconolite and a small amount of perovskite (P) is present.



**Figure 5:** SEM micrograph of sample #61 (5-Al) sintered at 1400°C. The matrix is 2M zirconolite and a small amount of Hf-  
rutile is present.

— 20  $\mu\text{m}$



**Figure 6:** SEM micrograph of sample #69 (1-Al) sintered at 1300°C. The matrix is pyrochlore and a trace of Gd/Al titanate  
(A) is present.

000498

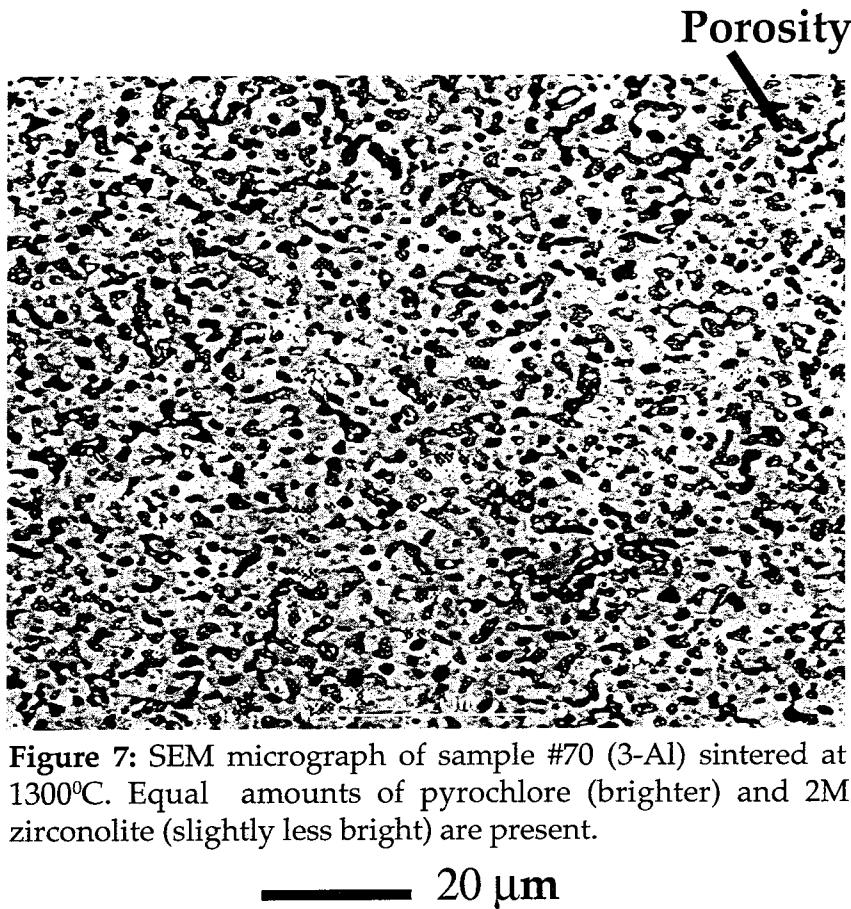


Figure 7: SEM micrograph of sample #70 (3-Al) sintered at 1300°C. Equal amounts of pyrochlore (brighter) and 2M zirconolite (slightly less bright) are present.

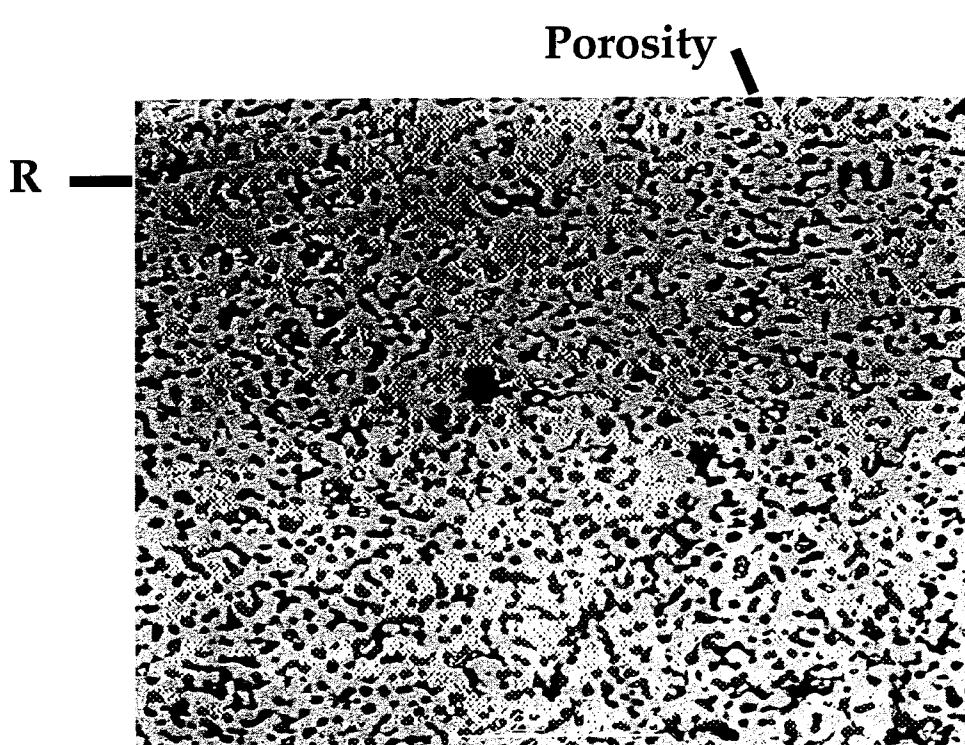


Figure 8: SEM micrograph of sample #71 (5-Al) sintered at 1300°C. The matrix is 2M zirconolite and a small amount of Hf-rutile (R) is present.

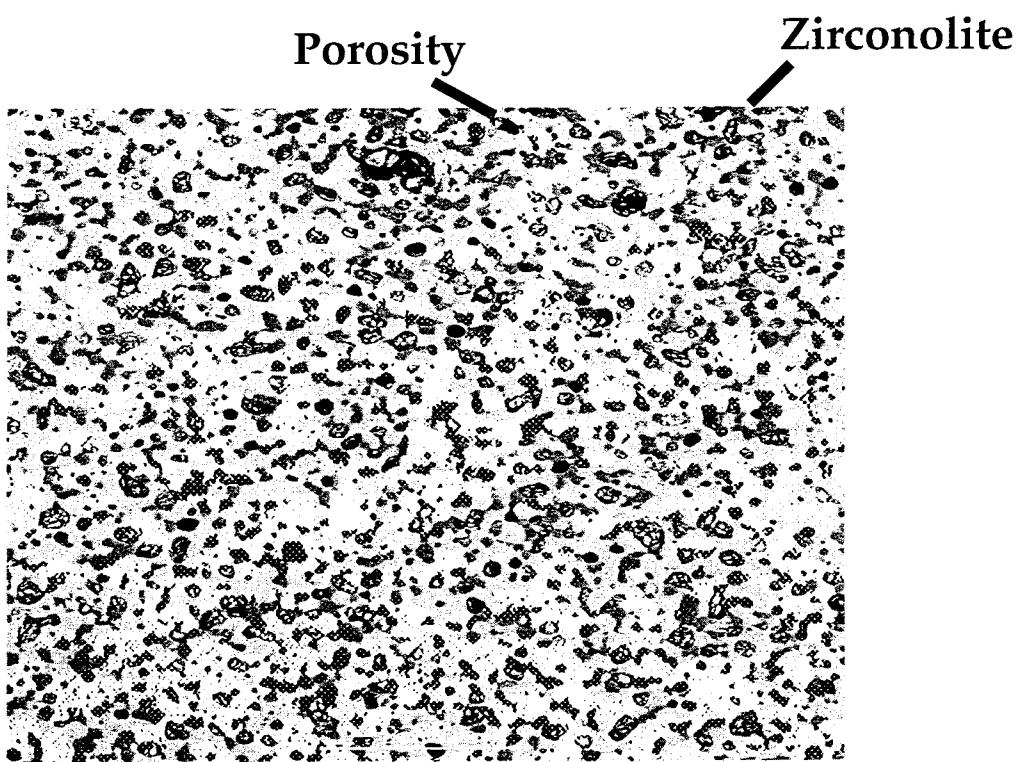


Figure 9: SEM micrograph of sample #74 (2-Al) sintered at 1350°C. The matrix is pyrochlore and 2M zirconolite is present.

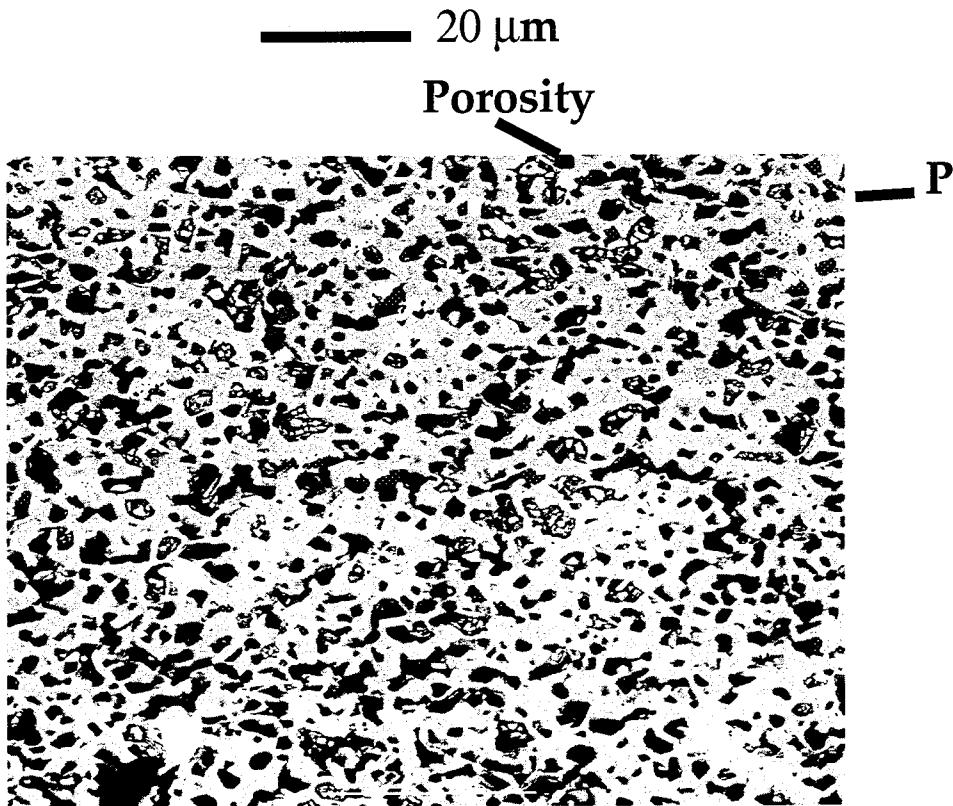


Figure 10: SEM micrograph of sample #75 (4-Al) sintered at 1350°C. The matrix is 2M zirconolite and a small amount of perovskite (P) is present.

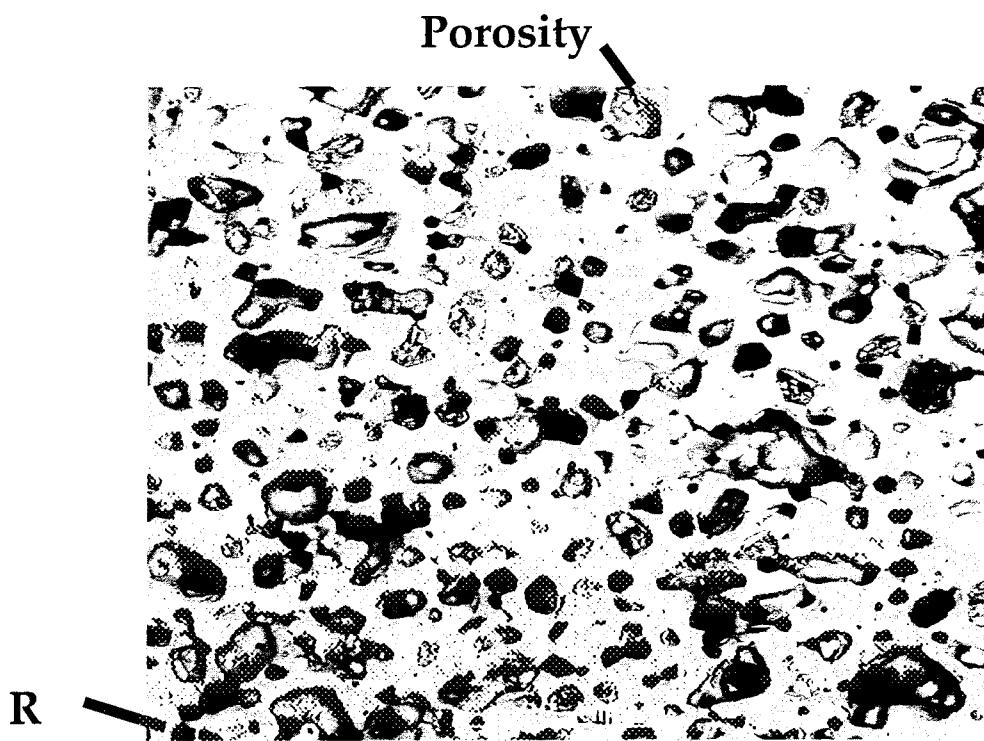


Figure 11: SEM micrograph of sample #62 (1-Mg) sintered at 1400°C. The matrix is pyrochlore and a small amount of rutile (R) is present.

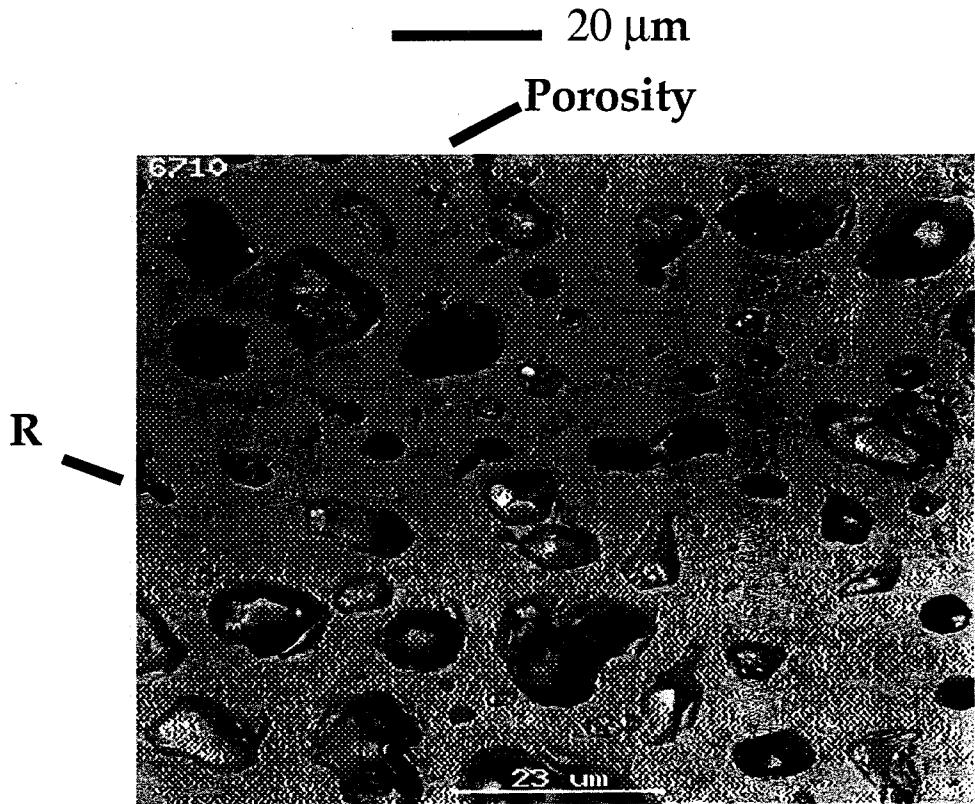


Figure 12: SEM micrograph of sample #63 (2-Mg) sintered at 1400°C. The matrix is pyrochlore and a small amount of Hf-rutile (R) is present.

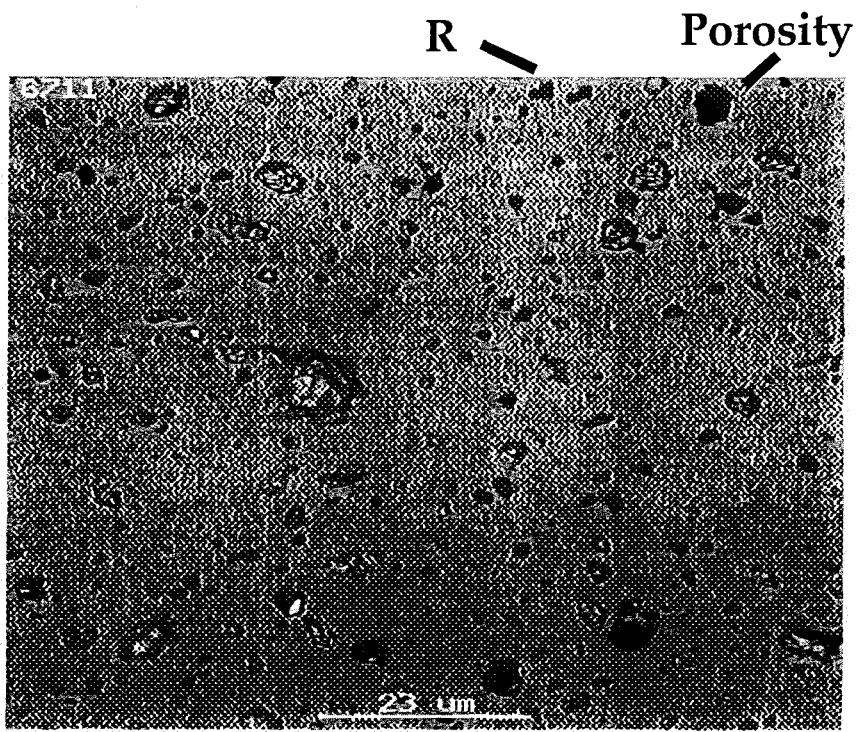


Figure 13: SEM micrograph of sample #64 (3-Mg) sintered at 1400°C. The matrix is 2M zirconolite and a trace of Hf-rutile (R) is present.

— 20  $\mu\text{m}$

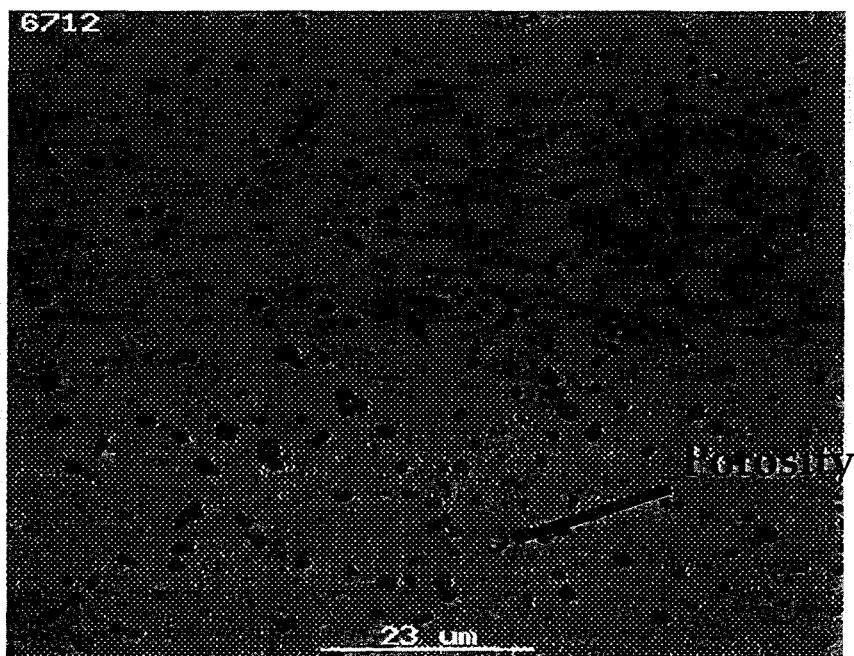


Figure 14: SEM micrograph of sample #65 (4-Mg) sintered at 1400°C. The matrix is 2M zirconolite and pyrochlore (see Fig. 15) with a small amount of Hf-rutile (dark grey)

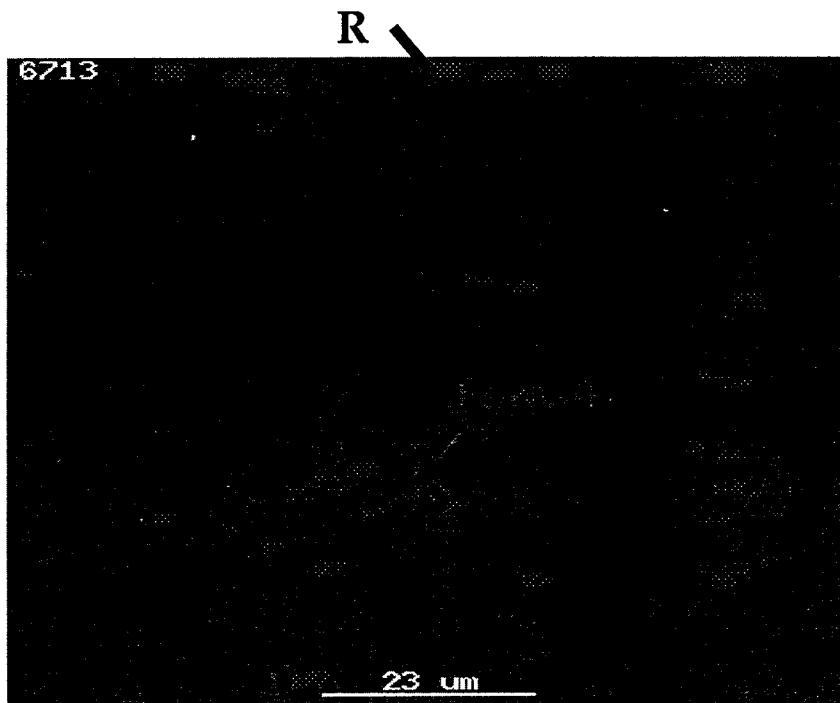
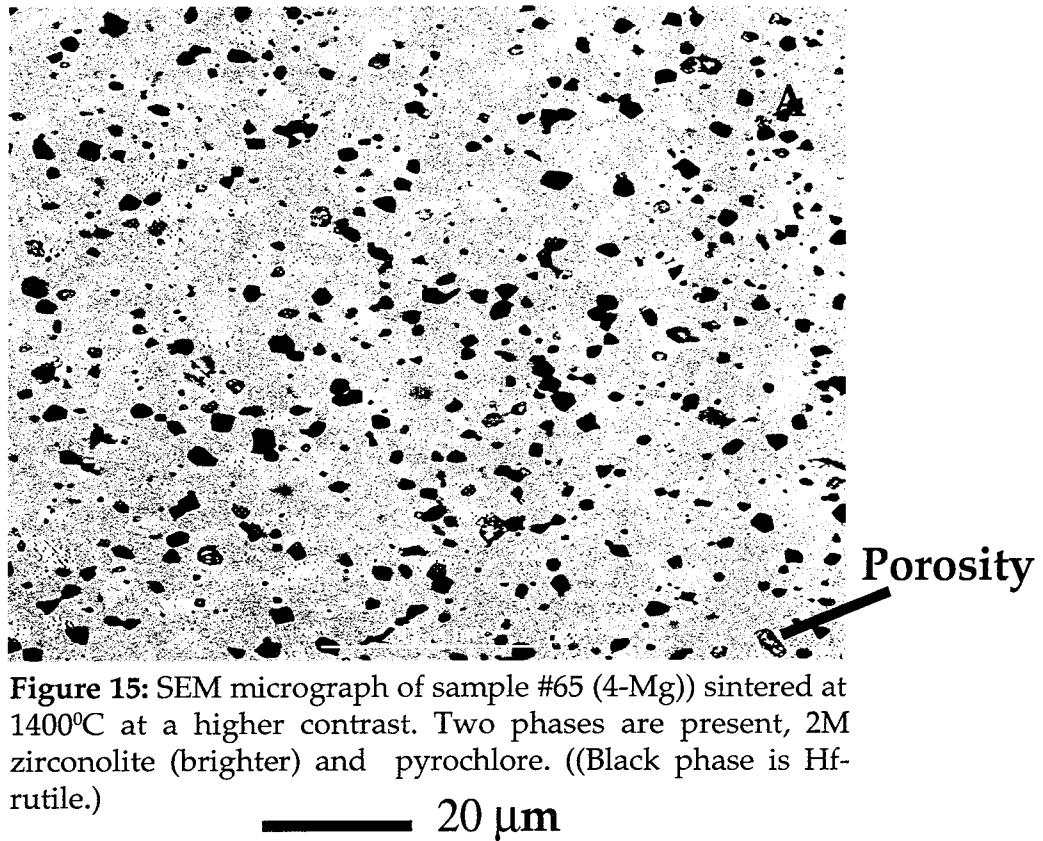


Figure 16: SEM micrograph of sample #66 (5-Mg) sintered at 1400°C. The matrix is 2M zirconolite with a trace of Hf-rutile.

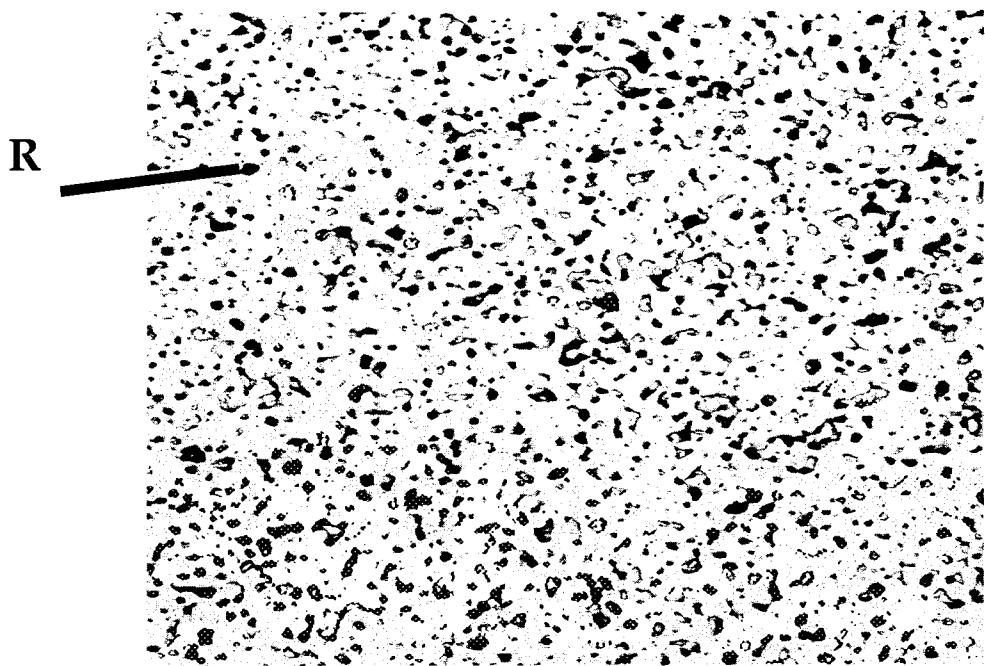


Figure 17: SEM micrograph of sample #90 (1-Mg) sintered at 1300°C. The matrix is pyrochlore and a small amount of rutile (R) is present.

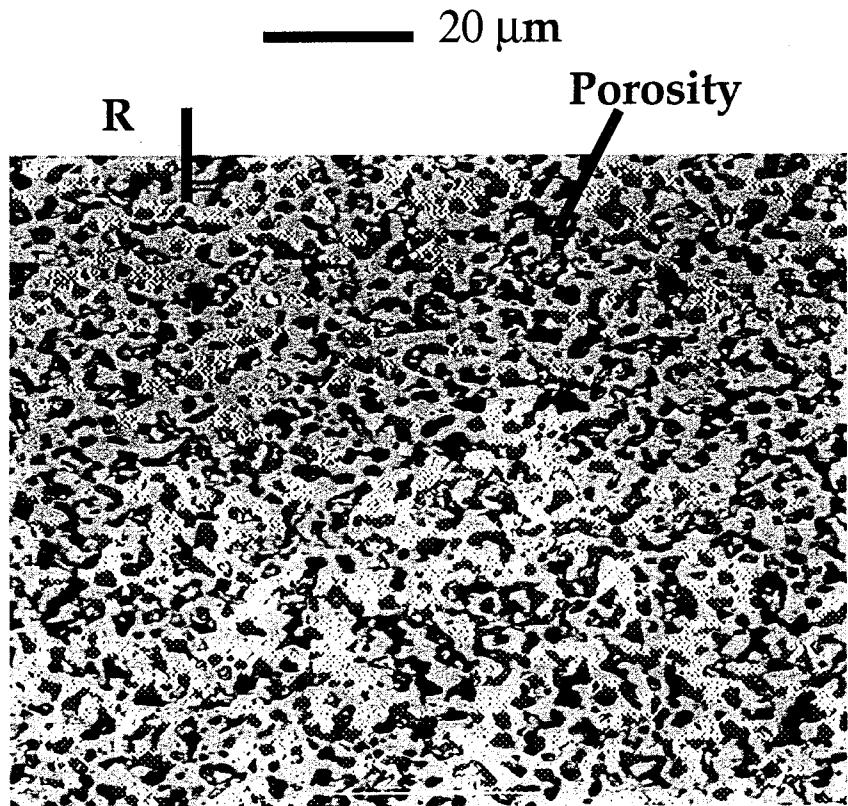


Figure 18: SEM micrograph of sample #91 (3-Mg) sintered at 1300°C. The matrix is 2M zirconolite and a small amount of Hf-rutile(R ) is present.

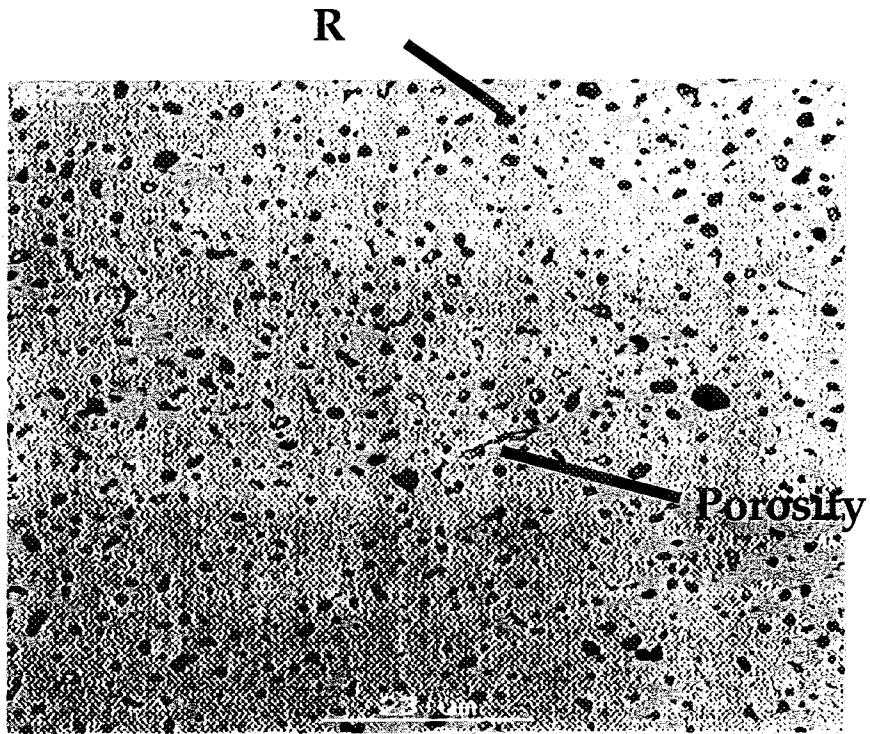


Figure 19: SEM micrograph of sample #92 (5-Mg) sintered at 1300°C. The matrix is 2M zirconolite and a small amount of Hf-rutile (R) is present.

— 20 μm

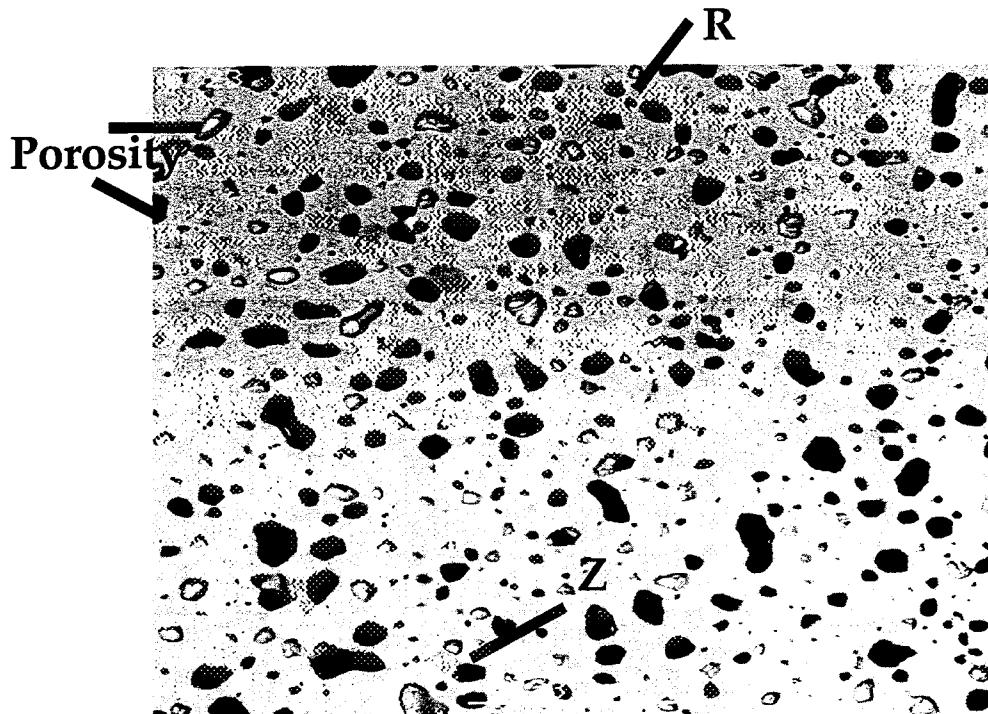
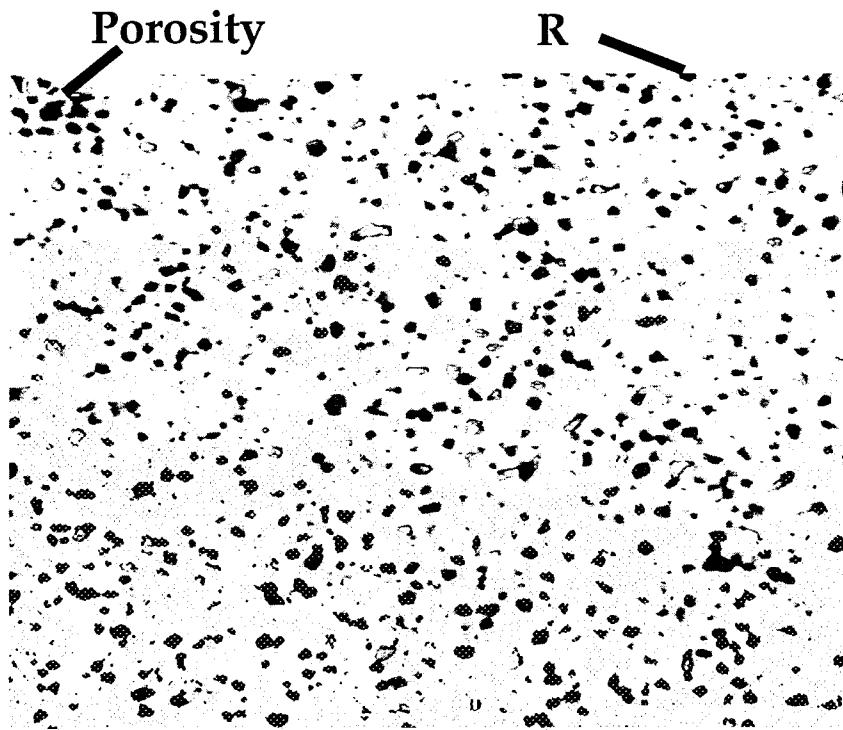
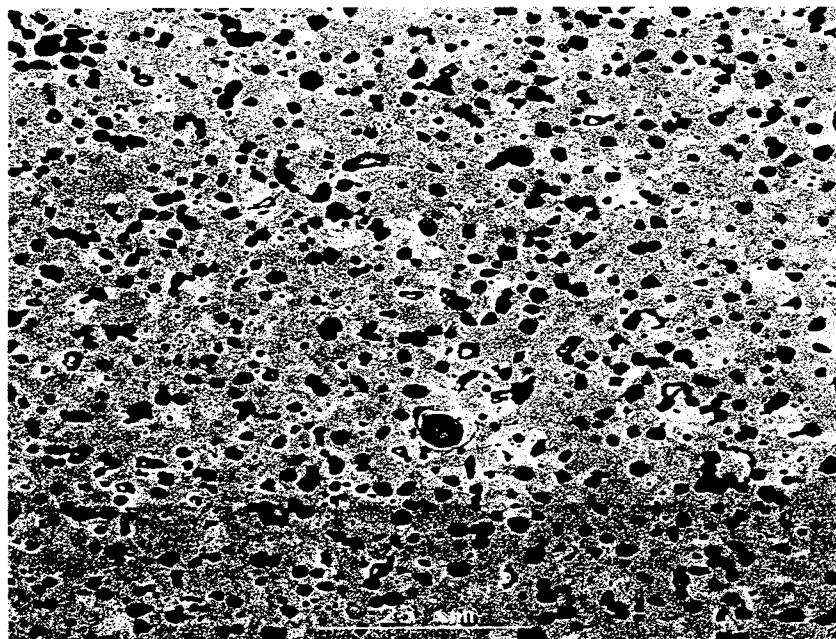


Figure 20: SEM micrograph of sample #93 (2-Mg) sintered at 1350°C. The matrix is pyrochlore and 2M zirconolite (Z). A trace of Hf-rutile (R) is present.



**Figure 21:** SEM micrograph of sample #94 (4-Mg) sintered at 1350°C. The matrix is pyrochlore and 2M zirconolite with a small amount of Hf-rutile (difficult to distinguish from the porosity). (See Fig. 22.)

20  $\mu\text{m}$



**Figure 22:** SEM micrograph of sample #94 (4-Mg) sintered at 1350°C at a higher contrast. The matrix is pyrochlore and 2M zirconolite (brighter). The porosity and Hf-rutile are indistinguishable.

## **Appendix 3**

### **EDS Analysis**

Tables 1-19

**Table 1.** EDS analysis of sample #57 (1-Al).

**L990657\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Wt % (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Gd-L	0.4831	1.217	17.35	58.81	+/- 0.47	Gd2O3	67.78	1.909
Al-K	0.0023	2.188	0.87	0.51	+/- 0.04	Al2O3	0.96	0.096
Ti-K	0.1916	0.978	18.15	18.74	+/- 0.12	TiO2	31.26	1.997
O -K	---	3.283	63.63	21.94 S	---	---	---	---
Total			100.00	100.00			100.00	4.001

The number of cation results are based upon 7 Oxygen atoms

**L990657\_Al-titanate**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Wt % (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Al-K	0.1736	1.562	24.19	27.12	+/- 0.15	Al2O3	51.24	1.936
Gd-L	0.0461	1.489	1.05	6.87	+/- 0.46	Gd2O3	7.92	0.084
Ti-K	0.2128	1.151	12.30	24.48	+/- 0.23	TiO2	40.84	0.985
O -K	---	4.174	62.46	41.53 S	---	---	---	---
Total			100.00	100.00			100.00	3.005

The number of cation results are based upon 5 Oxygen atoms

**Table 2. EDS analysis of sample #58 (2-Al).**

**L990658\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err. Compound	Compound
Ca-K	0.0167	0.971	1.81	1.62	+/- 0.05	CaO	2.27
Hf-M	0.0199	2.049	1.02	4.07	+/- 0.13	HfO <sub>2</sub>	4.80
Gd-L	0.4185	1.233	14.68	51.61	+/- 0.44	Gd <sub>2</sub> O <sub>3</sub>	59.48
Al-K	0.0023	2.096	0.80	0.48	+/- 0.06	Al <sub>2</sub> O <sub>3</sub>	0.92
Ti-K	0.1961	0.994	18.21	19.50	+/- 0.12	TiO <sub>2</sub>	32.53
O -K	---	3.535	63.48	22.71 S	---	---	---
Total			100.00	100.00		100.00	4.026

The number of cation results are based upon 7 Oxygen atoms

**L990658\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err. Compound	Compound
Ca-K	0.0201	0.999	1.91	2.00	+/- 0.05	CaO	2.80
Hf-M	0.0619	1.879	2.49	11.64	+/- 0.16	HfO <sub>2</sub>	13.72
Gd-L	0.2599	1.287	8.13	33.44	+/- 0.38	Gd <sub>2</sub> O <sub>3</sub>	38.55
Al-K	0.0252	1.867	6.67	4.71	+/- 0.07	Al <sub>2</sub> O <sub>3</sub>	8.90
Ti-K	0.2084	1.036	17.23	21.60	+/- 0.13	TiO <sub>2</sub>	36.02
O -K	---	3.951	63.56	26.61 S	---	---	---
Total			100.00	100.00		100.00	4.013

The number of cation results are based upon 7 Oxygen atoms

**Table 3.** EDS analysis of sample #59 (3-Al).

**L990659\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound Formula	Compound Wt %
Ca-K	0.0306	0.981	3.22	3.00	+/- 0.06	CaO	4.19
Hf-M	0.0485	1.930	2.26	9.36	+/- 0.15	HfO2	11.04
Gd-L	0.3452	1.249	11.82	43.11	+/- 0.41	Gd2O3	49.69
Al-K	0.0024	1.983	0.77	0.48	+/- 0.06	Al2O3	0.91
Ti-K	0.2026	1.011	18.44	20.49	+/- 0.12	TiO2	34.17
O -K	---	3.813	63.49	23.57 S	---	---	---
Total			100.00	100.00			100.00
							4.025

The number of cation results are based upon 7 Oxygen atoms

**L990659\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound Formula	Compound Wt %
Ca-K	0.0419	1.009	4.06	4.22	+/- 0.06	CaO	5.91
Hf-M	0.1225	1.702	4.50	20.86	+/- 0.17	HfO2	24.60
Gd-L	0.1835	1.288	5.79	23.63	+/- 0.34	Gd2O3	27.23
Al-K	0.0194	1.717	4.75	3.32	+/- 0.07	Al2O3	6.28
Ti-K	0.2052	1.051	17.35	21.57	+/- 0.13	TiO2	35.98
O -K	---	4.268	63.56	26.40 S	---	---	---
Total			100.00	100.00			100.00
							4.014

The number of cation results are based upon 7 Oxygen atoms

**Table 5.** EDS analysis of sample #61 (5-Al).

**L990661\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound Formula	Compound Wt %
					Wt % (1-Sigma)		No. of Cations
Ca-K	0.0733	1.021	7.09	7.49	+/- 0.07	CaO	10.47
Hf-M	0.2223	1.465	6.92	32.56	+/- 0.19	HfO2	38.40
Gd-L	0.0668	1.301	2.10	8.69	+/- 0.29	Gd2O3	10.02
Al-K	0.0101	1.514	2.16	1.53	+/- 0.06	Al2O3	2.90
Ti-K	0.2130	1.075	18.14	22.90	+/- 0.14	TiO2	38.20
O -K	---	4.772	63.60	26.82 S	---	---	---
Total			100.00	100.00			100.00
							4.007

The number of cation results are based upon 7 Oxygen atoms

**L990661\_Hf-rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound Formula	Compound Wt %
					Wt % (1-Sigma)		No. of Cations
Ca-K	0.0046	0.971	0.33	0.45	+/- 0.02	CaO	0.63
Hf-M	0.0901	1.471	2.19	13.26	+/- 0.11	HfO2	15.63
Gd-L	0.0026	1.429	0.07	0.37	+/- 0.11	Gd2O3	0.43
Al-K	0.0016	1.582	0.28	0.25	+/- 0.02	Al2O3	0.48
Ti-K	0.4554	1.090	30.63	49.66	+/- 0.22	TiO2	82.83
O -K	---	5.935	66.50	36.01 S	---	---	---
Total			100.00	100.00			100.00
							1.008

The number of cation results are based upon 2 Oxygen atoms

**Table 6.** EDS analysis of sample #69 (1-Al).

**L990669\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Wt % (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Gd-L	0.4834	1.217	17.37	58.84	+/- 0.63	Gd2O3	67.82	1.911
Al-K	0.0021	2.189	0.81	0.47	+/- 0.06	Al2O3	0.89	0.089
Ti-K	0.1918	0.978	18.19	18.76	+/- 0.16	TiO2	31.30	2.000
O -K	---	3.284	63.64	21.93 S	---	---	---	---
Total			100.00	100.00			100.00	4.000

The number of cation results are based upon 7 Oxygen atoms

**L990669\_Gd-Al\_titanate**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Wt % (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Gd-L	0.2298	1.353	6.57	31.09	+/- 0.48	Gd2O3	35.83	1.119
Al-K	0.0250	1.942	5.99	4.86	+/- 0.06	Al2O3	9.18	1.020
Ti-K	0.3147	1.048	22.87	32.96	+/- 0.20	TiO2	54.99	3.896
O -K	---	4.488	64.57	31.09 S	---	---	---	---
Total			100.00	100.00			100.00	6.035

The number of cation results are based upon 11 Oxygen atoms

**Table 7. EDS analysis of sample #71 (5-Al).**

**L990671\_zirconolite**

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err.	Compound	Compound	No. of Cations
							Wt %	(1-Sigma)	
Ca-K	0.0734	1.022	7.14	7.50	+/- 0.08		CaO	10.49	0.786
Hf-M	0.2271	1.460	7.09	33.16	+/- 0.21		HfO <sub>2</sub>	39.11	0.780
Gd-L	0.0663	1.298	2.09	8.61	+/- 0.33		Gd <sub>2</sub> O <sub>3</sub>	9.93	0.230
Al-K	0.0100	1.509	2.13	1.51	+/- 0.07		Al <sub>2</sub> O <sub>3</sub>	2.85	0.235
Ti-K	0.2099	1.075	17.97	22.55	+/- 0.15		TiO <sub>2</sub>	37.62	1.978
O -K	---	4.752	63.58	26.66 S	---	---	---	---	---
Total			100.00	100.00				100.00	4.009

The number of cation results are based upon 7 Oxygen atoms

**L990671\_rutile**

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err.	Compound	Compound	No. of Cations
							Wt %	(1-Sigma)	
Hf-M	0.0722	1.478	1.73	10.67	+/- 0.18		HfO <sub>2</sub>	12.58	0.052
Ti-K	0.4800	1.092	31.61	52.41	+/- 0.27		TiO <sub>2</sub>	87.42	0.948
O -K	---	6.072	66.67	36.92 S	---	---	---	---	---
Total			100.00	100.00				100.00	1.000

The number of cation results are based upon 2 Oxygen atoms

**Table 8. EDS analysis of sample #74 (2-Al).**

**L990674\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound Formula	Compound Wt %	No. of Cations
Ca-K	0.0147	0.971	1.59	1.43	+/- 0.06	CaO	1.99	0.175
Hf-M	0.0169	2.059	0.87	3.49	+/- 0.16	HfO2	4.11	0.096
Gd-L	0.4222	1.234	14.78	52.09	+/- 0.49	Gd2O3	60.04	1.629
Al-K	0.0027	2.105	0.95	0.58	+/- 0.07	Al2O3	1.09	0.105
Ti-K	0.1977	0.994	18.29	19.64	+/- 0.14	TiO2	32.76	2.016
O -K	---	3.522	63.52	22.78 S	---	---	---	---
Total			100.00	100.00			100.00	4.021

The number of cation results are based upon 7 Oxygen atoms

**L990674\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound Formula	Compound Wt %	No. of Cations
Ca-K	0.0218	1.003	2.06	2.19	+/- 0.06	CaO	3.06	0.228
Hf-M	0.0713	1.852	2.80	13.20	+/- 0.19	HfO2	15.57	0.309
Gd-L	0.2422	1.290	7.52	31.23	+/- 0.42	Gd2O3	36.00	0.828
Al-K	0.0278	1.833	7.14	5.09	+/- 0.08	Al2O3	9.61	0.787
Ti-K	0.2060	1.041	16.94	21.44	+/- 0.14	TiO2	35.76	1.867
O -K	---	3.990	63.54	26.85 S	---	---	---	---
Total			100.00	100.00			100.00	4.017

The number of cation results are based upon 7 Oxygen atoms

**Table 9.** EDS analysis of sample #75 (4-Al).

**L99067\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound (1-Sigma)	Compound Formula	No. of Cations
					Wt %		Wt %	
Ca-K	0.0515	1.005	5.12	5.17	+/- 0.07	CaO	7.23	0.564
Hf-M	0.1298	1.680	4.85	21.80	+/- 0.20	HfO2	25.71	0.534
Gd-L	0.1884	1.280	6.09	24.12	+/- 0.39	Gd2O3	27.81	0.671
Al-K	0.0097	1.722	2.46	1.67	+/- 0.07	Al2O3	3.16	0.271
Ti-K	0.2065	1.048	17.94	21.64	+/- 0.14	TiO2	36.10	1.977
O -K	---	4.333	63.53	25.60 S	---	---	---	---
Total			100.00	100.00			100.00	4.018

The number of cation results are based upon 7 Oxygen atoms

**L99067\_perovskite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound (1-Sigma)	Compound Formula	No. of Cations
					Wt %		Wt %	
Ca-K	0.1511	0.989	12.44	14.95	+/- 0.10	CaO	20.91	0.608
Hf-M	0.0116	1.794	0.39	2.09	+/- 0.13	HfO2	2.46	0.019
Gd-L	0.1810	1.379	5.30	24.96	+/- 0.39	Gd2O3	28.77	0.259
Al-K	0.0058	1.868	1.34	1.09	+/- 0.06	Al2O3	2.05	0.066
Ti-K	0.2501	1.098	19.12	27.45	+/- 0.16	TiO2	45.79	0.934
O -K	---	5.417	61.41	29.46 S	---	---	---	---
Total			100.00	100.00			100.00	1.885

The number of cation results are based upon 3 Oxygen atoms

**Table 10.** EDS analysis of sample #62 (1-Mg).

**L990662\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Wt % (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Gd-L	0.4610	1.229	16.20	56.63	+/- 0.50	Gd2O3	65.27	1.789
Mg-K	0.0033	2.884	1.74	0.94	+/- 0.13	MgO	1.55	0.192
Ti-K	0.2021	0.984	18.68	19.89	+/- 0.13	TiO2	33.17	2.062
O -K	---	3.374	63.39	22.54 S	---	---	---	---
Total			100.00	100.00			100.00	4.043

The number of cation results are based upon 7 Oxygen atoms

**L990662\_rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Wt % (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Hf-M	0.0006	1.535	0.01	0.09	+/- 0.04	HfO2	0.10	0.000
Ti-K	0.5429	1.103	33.32	59.89	+/- 0.28	TiO2	99.89	1.000
O -K	---	6.543	66.67	40.02 S	---	---	---	---
Total			100.00	100.00			100.00	1.000

The number of cation results are based upon 2 Oxygen atoms

**Table 11.** EDS analysis of sample #63 (2-Mg).

**L990663\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	Compound	Compound	No. of Cations
					Wt %	Formula	Wt %	
Ca-K	0.0175	0.976	1.85	1.71	+/- 0.06	CaO	2.39	0.204
Hf-M	0.0300	2.000	1.46	5.99	+/- 0.11	HfO <sub>2</sub>	7.07	0.161
Gd-L	0.3815	1.244	13.11	47.47	+/- 0.47	Gd <sub>2</sub> O <sub>3</sub>	54.72	1.448
Mg-K	0.0032	2.684	1.55	0.87	+/- 0.12	MgO	1.43	0.171
Ti-K	0.2057	1.003	18.69	20.62	+/- 0.14	TiO <sub>2</sub>	34.39	2.065
O -K	---	3.663	63.35	23.34 S	---	---	---	---
Total			100.00	100.00			100.00	4.050

The number of cation results are based upon 7 Oxygen atoms

**L990663\_rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg								
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	Compound	Compound	No. of Cations
					Wt %	Formula	Wt %	
Hf-M	0.0430	1.500	1.01	6.45	+/- 0.08	HfO <sub>2</sub>	7.60	0.030
Ti-K	0.5052	1.096	32.32	55.38	+/- 0.25	TiO <sub>2</sub>	92.38	0.970
O -K	---	6.261	66.66	38.16 S	---	---	---	---
Total			100.00	100.00			100.00	1.000

The number of cation results are based upon 2 Oxygen atoms

**Table 12.** EDS analysis of sample #64 (3-Mg).

**L990664\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg									
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err. (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Ca-K	0.0356	0.990	3.65	3.53	+/- 0.16	Cao	4.94	0.401	
Hf-M	0.0852	1.798	3.56	15.32	+/- 0.31	HfO2	18.06	0.391	
Gd-L	0.2661	1.265	8.87	33.66	+/- 0.63	Gd2O3	38.79	0.975	
Mg-K	0.0018	2.387	0.74	0.43	+/- 0.10	MgO	0.72	0.081	
Ti-K	0.2189	1.027	19.45	22.47	+/- 0.31	TiO2	37.49	2.137	
O -K	---	4.111	63.72	24.59 S	---	---	---	---	---
Total			100.00	100.00			100.00	3.985	

The number of cation results are based upon 7 Oxygen atoms

**L990664\_rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg									
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err. (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Hf-M	0.0629	1.485	1.50	9.34	+/- 0.11	HfO2	11.02	0.045	
Ti-K	0.4880	1.093	31.84	53.35	+/- 0.28	TiO2	88.98	0.955	
O -K	---	6.132	66.67	37.31 S	---	---	---	---	---
Total			100.00	100.00			100.00	1.000	

The number of cation results are based upon 2 Oxygen atoms

**Table 13.** EDS analysis of sample #65 (4-Mg).

**L990665\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	Compound Formula	Compound Wt % No. of Cations
Ca-K	0.0480	1.001	4.86	4.81	+/- 0.07	CaO	6.73 0.537
Hf-M	0.1308	1.679	4.99	21.97	+/- 0.15	HfO2	25.91 0.551
Gd-L	0.1985	1.273	6.52	25.28	+/- 0.40	Gd2O3	29.13 0.719
Mg-K	0.0043	2.190	1.58	0.95	+/- 0.09	MgO	1.57 0.175
Ti-K	0.2109	1.042	18.61	21.98	+/- 0.14	TiO2	36.66 2.054
O -K	---	4.307	63.43	25.02 S	---	---	---
Total			100.00	100.00		100.00	4.036

The number of cation results are based upon 7 Oxygen atoms

**L990665\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	Compound Formula	Compound Wt % No. of Cations
Ca-K	0.0484	1.010	4.95	4.89	+/- 0.12	CaO	6.84 0.545
Hf-M	0.1830	1.575	6.55	28.83	+/- 0.24	HfO2	33.99 0.721
Gd-L	0.1454	1.272	4.77	18.49	+/- 0.62	Gd2O3	21.31 0.525
Mg-K	0.0058	2.021	1.94	1.16	+/- 0.14	MgO	1.93 0.214
Ti-K	0.2052	1.049	18.22	21.53	+/- 0.22	TiO2	35.92 2.006
O -K	---	4.381	63.58	25.09 S	---	---	---
Total			100.00	100.00		100.00	4.011

The number of cation results are based upon 7 Oxygen atoms

**L990665\_rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. (1-Sigma)	Compound Formula	Compound Wt % No. of Cations
Hf-M	0.1065	1.453	2.60	15.47	+/- 0.11	HfO2	18.24 0.078
Ti-K	0.4510	1.087	30.73	49.01	+/- 0.24	TiO2	81.75 0.922
O -K	---	5.855	66.67	35.52 S	---	---	---
Total			100.00	100.00		100.00	1.000

The number of cation results are based upon 2 Oxygen atoms

**Table 14.** EDS analysis of sample #66 (5-Mg).

**L990666\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg						
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound
				Wt %	(1-Sigma)	Formula
Ca-K	0.0751	1.018	7.21	7.64	+/- 0.08	CaO
Hf-M	0.2169	1.458	6.70	31.62	+/- 0.16	HfO2
Gd-L	0.0660	1.306	2.07	8.61	+/- 0.32	Gd2O3
Mg-K	0.0036	1.855	1.05	0.68	+/- 0.07	MgO
Ti-K	0.2285	1.075	19.40	24.57	+/- 0.16	TiO2
O -K	---	4.879	63.57	26.89 S	---	---
Total			100.00	100.00		100.00
						4.012

The number of cation results are based upon 7 Oxygen atoms

**L990666\_rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg						
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound
				Wt %	(1-Sigma)	Formula
Hf-M	0.0901	1.464	2.18	13.19	+/- 0.16	HfO2
Ti-K	0.4648	1.089	31.16	50.63	+/- 0.26	TiO2
O -K	---	5.958	66.67	36.18 S	---	---
Total			100.00	100.00		100.00
						1.000

The number of cation results are based upon 2 Oxygen atoms

**Table 15.** EDS analysis of sample #90 (1-Mg).

**L990690\_pyrochlore**

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err.	Compound	Compound	No. of Cations
							Wt %	(1-Sigma)	
Gd-L	0.4610	1.228	16.22	56.63	+/- 0.52		Gd2O3	65.28	1.789
Mg-K	0.0028	2.885	1.51	0.81	+/- 0.14		MgO	1.35	0.166
Ti-K	0.2034	0.984	18.81	20.01	+/- 0.14		TiO2	33.38	2.075
O -K	---	3.380	63.46	22.54 S	---	---	---	---	---
Total			100.00	100.00				100.00	4.030

The number of cation results are based upon 7 Oxygen atoms

**L990690\_rutile**

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err.	Compound	Compound	No. of Cations
							Wt %	(1-Sigma)	
Mg-K	0.0000	2.076	0.00	0.00	+/- 0.00		MgO	0.00	0.000
Ti-K	0.5434	1.103	33.33	59.95	+/- 0.28		TiO2	100.00	1.000
O -K	---	6.547	66.67	40.05 S	---	---	---	---	---
Total			100.00	100.00				100.00	1.000

The number of cation results are based upon 2 Oxygen atoms

000496

**Table 16.** EDS analysis of sample #91 (3-Mg).**L990691\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Compound	Compound	No. of Cations
				Wt %	(1-Sigma)	Formula	Wt %
Ca-K	0.0353	0.992	3.57	3.50	+/- 0.07	CaO	4.90
Hf-M	0.0812	1.811	3.37	14.70	+/- 0.14	HfO <sub>2</sub>	17.33
Gd-L	0.2654	1.269	8.76	33.67	+/- 0.44	Gd <sub>2</sub> O <sub>3</sub>	38.81
Mg-K	0.0055	2.387	2.19	1.30	+/- 0.11	MgO	2.16
Ti-K	0.2143	1.029	18.83	22.06	+/- 0.15	TiO <sub>2</sub>	36.79
O -K	---	4.088	63.29	24.77 S	---	---	---
Total			100.00	100.00			100.00
							4.061

The number of cation results are based upon 7 Oxygen atoms

**L990691\_rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg							
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err. Compound	Compound	No. of Cations
				Wt %	(1-Sigma)	Formula	Wt %
Hf-M	0.0575	1.489	1.36	8.56	+/- 0.11	HfO <sub>2</sub>	10.10
Ti-K	0.4927	1.094	31.97	53.90	+/- 0.27	TiO <sub>2</sub>	89.90
O -K	---	6.167	66.67	37.54 S	---	---	---
Total			100.00	100.00			100.00
							1.000

The number of cation results are based upon 2 Oxygen atoms

000496

**Table 17.** EDS analysis of sample #92 (5-Mg).**L990692\_zirconolite**

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err. (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Ca-K	0.0745	1.018	7.15	7.58	+/- 0.09		CaO	10.61	0.787
Hf-M	0.2187	1.455	6.74	31.82	+/- 0.17		HfO <sub>2</sub>	37.53	0.742
Gd-L	0.0645	1.306	2.02	8.42	+/- 0.34		Gd <sub>2</sub> O <sub>3</sub>	9.70	0.223
Mg-K	0.0040	1.850	1.15	0.74	+/- 0.08		MgO	1.23	0.127
Ti-K	0.2283	1.075	19.37	24.54	+/- 0.16		TiO <sub>2</sub>	40.94	2.133
O -K	---	4.876	63.56	26.90 S	---	---	---	---	---
Total			100.00	100.00				100.00	4.013

The number of cation results are based upon 7 Oxygen atoms

**L990692\_rutile**

Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt %	Err. (1-Sigma)	Compound Formula	Compound Wt %	No. of Cations
Hf-M	0.0793	1.472	1.90	11.67	+/- 0.11		HfO <sub>2</sub>	13.77	0.057
Ti-K	0.4739	1.091	31.43	51.70	+/- 0.22		TiO <sub>2</sub>	86.23	0.943
O -K	---	6.026	66.67	36.63 S	---	---	---	---	---
Total			100.00	100.00				100.00	1.000

The number of cation results are based upon 2 Oxygen atoms

**Table 18.** EDS analysis of sample #93 (2-Mg).**L990693\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg									
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound	Compound	No. of Cations	
					Wt %	(1-Sigma)	Formula	Wt %	
Ca-K	0.0175	0.977	1.85	1.71	+/- 0.06		CaO	2.39	0.204
Hf-M	0.0305	1.999	1.48	6.10	+/- 0.12		HfO <sub>2</sub>	7.19	0.164
Gd-L	0.3790	1.245	12.98	47.20	+/- 0.49		Gd <sub>2</sub> O <sub>3</sub>	54.40	1.437
Mg-K	0.0041	2.678	1.94	1.09	+/- 0.13		MgO	1.80	0.214
Ti-K	0.2044	1.004	18.52	20.51	+/- 0.14		TiO <sub>2</sub>	34.21	2.049
O -K	---	3.661	63.24	23.40 S	---	---	---	---	---
Total			100.00	100.00				100.00	4.069

The number of cation results are based upon 7 Oxygen atoms

**L990693\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg									
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound	Compound	No. of Cations	
					Wt %	(1-Sigma)	Formula	Wt %	
Ca-K	0.0182	0.997	1.81	1.82	+/- 0.06		CaO	2.54	0.200
Hf-M	0.0931	1.786	3.72	16.63	+/- 0.15		HfO <sub>2</sub>	19.61	0.411
Gd-L	0.2409	1.274	7.78	30.68	+/- 0.44		Gd <sub>2</sub> O <sub>3</sub>	35.36	0.861
Mg-K	0.0116	2.315	4.39	2.68	+/- 0.11		MgO	4.44	0.486
Ti-K	0.2212	1.031	19.00	22.81	+/- 0.15		TiO <sub>2</sub>	38.05	2.101
O -K	---	4.054	63.30	25.39 S	---	---	---	---	---
Total			100.00	100.00				100.00	4.058

The number of cation results are based upon 7 Oxygen atoms

**L990693\_rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg									
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound	Compound	No. of Cations	
					Wt %	(1-Sigma)	Formula	Wt %	
Hf-M	0.0401	1.502	0.94	6.02	+/- 0.10		HfO <sub>2</sub>	7.10	0.028
Ti-K	0.5079	1.097	32.39	55.69	+/- 0.24		TiO <sub>2</sub>	92.90	0.972
O -K	---	6.281	66.67	38.28 S	---	---	---	---	---
Total			100.00	100.00				100.00	1.000

The number of cation results are based upon 2 Oxygen atoms

**Table 19.** EDS analysis of sample #94 (4-Mg).**L990694\_pyrochlore**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg									
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound	Compound	No. of Cations	
					Wt %	(1-Sigma)	Formula	Wt %	
Ca-K	0.0478	1.003	4.87	4.79	+/- 0.08		CaO	6.71	0.537
Hf-M	0.1413	1.660	5.35	23.45	+/- 0.16		HfO <sub>2</sub>	27.65	0.590
Gd-L	0.1900	1.271	6.25	24.15	+/- 0.43		Gd <sub>2</sub> O <sub>3</sub>	27.84	0.690
Mg-K	0.0046	2.158	1.65	0.98	+/- 0.10		MgO	1.63	0.182
Ti-K	0.2079	1.043	18.43	21.68	+/- 0.15		TiO <sub>2</sub>	36.17	2.033
O -K	---	4.305	63.45	24.94 S	---	---	---	---	---
Total			100.00	100.00				100.00	4.032

The number of cation results are based upon 7 Oxygen atoms

**L990694\_zirconolite**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg									
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound	Compound	No. of Cations	
					Wt %	(1-Sigma)	Formula	Wt %	
Ca-K	0.0480	1.012	4.97	4.86	+/- 0.08		CaO	6.80	0.548
Hf-M	0.1958	1.560	7.01	30.54	+/- 0.18		HfO <sub>2</sub>	36.02	0.772
Gd-L	0.1406	1.266	4.64	17.81	+/- 0.40		Gd <sub>2</sub> O <sub>3</sub>	20.53	0.511
Mg-K	0.0063	1.993	2.10	1.25	+/- 0.09		MgO	2.07	0.231
Ti-K	0.1977	1.049	17.74	20.74	+/- 0.15		TiO <sub>2</sub>	34.59	1.955
O -K	---	4.340	63.54	24.81 S	---	---	---	---	---
Total			100.00	100.00				100.00	4.017

The number of cation results are based upon 7 Oxygen atoms

**L990694\_rutile**

PROZA Correction Acc.Volt.= 15 kV Take-off Angle=30.00 deg									
Element	k-ratio (calc.)	ZAF	Atom %	Element	Wt % Err.	Compound	Compound	No. of Cations	
					Wt %	(1-Sigma)	Formula	Wt %	
Hf-M	0.1046	1.454	2.55	15.22	+/- 0.12		HfO <sub>2</sub>	17.94	0.077
Ti-K	0.4526	1.087	30.78	49.19	+/- 0.25		TiO <sub>2</sub>	82.06	0.923
O -K	---	5.866	66.67	35.59 S	---	---	---	---	---
Total			100.00	100.00				100.00	1.000

The number of cation results are based upon 2 Oxygen atoms